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Glassblowing for Laboratory Technicians

(including vacuum line accessories and their applications)

BY

R. BARBOUR

Member of the New Zealand Institute of Science Technicians (Inc.)

SECOND EDITION

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Foreword

PRACTICALLY every scientist must, with greater or lesser frequency, use laboratory glassware. Most have attempted at least some of the elementary operations of glassworking. Both experiences lead him to an awareness of those qualities of glass which, on the one hand, place at his disposal artefacts combining indispensable utility with considerable aesthetic appeal, and, on the other, require for the fabrication of these tools the skill of the craftsman fashioning an intractable material. The nature of this craft is such that nothing will replace the relationship of master and apprentice, and I have observed with admiration the effectiveness of Mr. Barbour's work in training young people, and particularly in helping them to an early feeling of confidence in their capacity to achieve ultimate success. As he truly says: "Glassblowing ... is an art, and mastery of an art needs courage and an adventurous spirit. Art also demands from those who take it up a measure of humility and dedication." Today, however, there is need for increasing depth of knowledge and skill in design to marry art to the service of experimental science, and in the more advanced chapters the author brings a wealth of experience to the writing, particularly of the sections on vacuum technique and interchangeable ground-glass joints.

It is not easy to describe the techniques of glassworking or to acquire them from the reading of the printed word, but no beginner can fail to benefit from the wealth of practical information, set out in meticulous detail, contained in the earlier chapters and fully illustrated by the accompanying plates. Similarly, those conducting classes in schools and colleges will find much sage advice in the chapter on the organization of glassblowing classes. Finally, for those who may have doubts in this machine age on the professional future and rewards for the technician–

craftsman, it may be comforting to savour the author's sad cynicism, rooted in a lifetime's experience in teaching institutions—"About half the burettes used in the elementary chemistry laboratory will be damaged in some way in the course of an academic year."

The number of works devoted exclusively to scientific glassblowing must be quite small, and it is therefore an added and special pleasure to write this brief foreword and to commend this book to all who may be concerned with either the training of glassblowers or with glassblowing.

December 1965

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Preface to the First Edition

MODERN teaching, research and industrial laboratories are engaged in work that necessitates the use of glass apparatus. The great bulk of this glassware is purchased from laboratory furnishers and, whenever possible, they are the best and most economical sources of supply.

Science, however, is never static. New and modified methods are continually being introduced and the experimental apparatus involved must be made, modified and maintained. To meet part of this need there has appeared on the broad vista of scientific endeavour the figure of the laboratory glassblower. Those who are privileged to read scientific papers, reports and theses, will have no doubts that the glassblower's service to science is an important and valuable one.

To be fully effective a glassblower must be familiar with the possibilities and limitations of glass. He must be able and willing to discuss apparatus design with scientists, to contribute his knowledge and experience to the discussions, to tackle techniques that are new or difficult, and to attempt, where necessary, seemingly impossible tasks.

The early chapters of this book cover fully the glassblowing requirements of the City and Guilds of London Institute syllabus 119 for laboratory technician's work.

They will also serve to introduce to students and other laboratory workers the important first steps in glassblowing techniques.

For the benefit of those taking examinations it is recommended that in glassblowing the student should aim to reach a standard of achievement higher than that required by the syllabus.

A well-prepared student will enjoy meeting the challenge of

an examination. At the other end of the scale, an ill-prepared student will be worried by the prospect of having to do work that has been but partly mastered, and harassed in the examination room if he is faced by tasks he has never previously performed.

Subsequent chapters are intended for those technicians who have acquired an interest in glassworking, and who have the opportunity to apply their skill to making more complex glassware.

It is worth noting that, in the writer's experience, laboratories that indulge in the luxury of a glassblower invariably increase the scope and tempo of their research work. Within a short time the part-time glassblower becomes a full-time glassblower, and the full-time glassblower becomes a very busy man.

The attention of the young lady who desires to take up glassblowing, either as a career or as a required subject for a technician's certificate, is drawn to the competency of the large numbers of lady glassblowers employed during the war years. There is no doubt that their numbers have increased considerably in the post-war period.

The writer fully acknowledges his debt to many sources of information: to books on glassblowing; glass-tube manufacturers' information circulars; laboratory furnishers' catalogues; to many glassblowers (some mature in wisdom and skill, some fresh and enthusiastic learners); to professional scientists, some no longer with us, for making available their theoretical knowledge and for making demands without which interest and progress would have atrophied.

The writer expresses his thanks to all those members of the staff of Victoria University of Wellington, New Zealand, who contributed to this work by giving their time to discussions and criticisms of the text. Special credit is due to B. C. Walsh, M.A., of the English Department, for his critical and invaluable reading of the drafts; to R. F. Gledhill of the Glassblowing Department, who devoted much care and patience to the preparation of the sketches and made many helpful suggestions; to M. D. King for the photography; to I. Crichton, the apprentice

glassblower, who performed many of the basic techniques to the instructions given in the text and helped to make them effective.

The writer acknowledges the prompt response to a request for information on fuel gases made by Professor R. W. Douglas of Sheffield University; the co-operation given by the officers of New Zealand Industrial Gases, who supplied data on liquid petroleum gases and who made their firm's products available, free of charge. He is indebted to Mr. K. Guy, F.I.S.T., of University of Natal, who suggested the preparation of this book and who has given much sound advice and guidance; to Professor S. N. Slater of the Chemistry Department, Victoria University of Wellington, for his encouragement during the preparation of the manuscript, and for his kindness in writing the foreword.

Finally, credit is due to Miss Rita Watts. She undertook to decipher and type the manuscript when she had very little time to spare.

Wellington, New Zealand
December 1965

R. BARBOUR

Preface to the Second Edition

THIS is a reprint of the first edition except that some errors have been corrected and the section on refrigerants has been brought up to date. Parts of a few chapters have been rewritten in the hope that ambiguities have been removed.

In 1970 the author moved to a new base in the University of Waikato, Hamilton, New Zealand. Here the glassblowing workshop services the School of Science, composed of Earth Sciences, Biological Sciences, Physics and Chemistry. Foreseeably Chemistry, which teaches organic, inorganic, physical and stable isotope chemistry, and in recent years, biochemistry, has made the greatest demand on the service.

The teaching staff in the School, drawn from Britain, America, Australia, Africa, Germany, Canada and from most of the other universities in New Zealand, brought with them a broad experience of glassware and accessories together with an impressive willingness to innovate and to develop new apparatus incorporating a wider range of materials than were previously available in New Zealand.

Since the first edition attracted comments from readers beyond those engaged in technical work, it seems timely to include an appendix to the second edition in which some of the glassware, recently developed, is described. A list of the manufacturers and suppliers of accessories using the new materials is included.

The author is pleased to express thanks to staff members in the School of Science for help in the preparation of this edition. In particular Professor J. D. McCraw, Dean of Science, arranged for draughting and typing facilities to be available and gave unobtrusive support throughout the exercise. A. T. Wilson, Professor of Chemistry, whose need for novel glassware has influenced glassblowing in New Zealand for two decades,

stimulated many discussions on the removal of water from gas mixtures, and on the need for safety-consciousness in vacuum line laboratories. Dr. K. M. Mackay and Ann Mackay, answered many queries on the choice of words and their arrangement and made valuable comments on the draft of the manuscript and on units and measurements. Dr. A. Langdon discussed constant volume manometers and made useful suggestions. Mr. F. Bailey drew the figures in the appendix from very rough and disproportionate sketches.

Mrs. Margaret McLean and Mrs. Elaine Norton typed the manuscript and the repeats, corrected the errors and inserted the omissions.

Thanks are also due to Pergamon Press whose officers worked hard to make the first edition attractive and to ensure its widespread acceptance.

To all those who reviewed the first edition, set out their comments and drew attention to errors, gratitude is expressed. The criticisms were stimulating, the praise was always heart warming.

October 1977 R. BARBOUR

CHAPTER 1

Glass

GLASS is a man-made material, and the date of its invention can only be estimated, but there is some evidence that it was first made about 4000 years ago.

There are many books on the art of glassmaking available in technical and other libraries. They are interesting and informative and their perusal will add much to background knowledge.

To make glass, accurately weighed quantities of finely ground materials are thoroughly mixed together. The carefully selected constituents are then melted in a refractory pot. When the glass is homogeneous, free from bubbles and foreign matter, it is shaped, while molten, by drawing, pressing, moulding or blowing into the desired shape. It should be noted that glass cannot be extruded through dies, as metals and plastic materials are extruded, since hot glass would adhere to hot dies and would be cooled and solidified by cold dies.

The tubes and rods used in the laboratory are made by one of three methods. The first, and oldest, method requires a team of skilled men, known as primary glassblowers. One member of the team uses an iron tube, approximately 5 ft long and fitted with a mouthpiece at one end and a small flange at the other end. The flanged end of this blow-pipe, as it is called, is repeatedly dipped into the molten glass and turned continuously until a gob of the desired size has been formed.

The blow-pipe is now passed to another member of the team. He shapes the gob by skilful manipulation and then blows an air bubble into the molten glass.

A third man now attaches the previously heated flange of a 5 ft metal rod to the end of the prepared glass. These two men

move slowly away from one another, walking backwards, rotating the molten glass and stretching it into a tube. A high standard of skill is required to make a tube of uniform wall-thickness, to control the diameter by blowing and to achieve the desired dimensions just as the glass sets.

The newly drawn out tube is now laid on wooden laths, cut up into 5 ft (1·5 m) lengths which are annealed in an oven and sent to the packing department.

This team of men can produce tubing and rod of any desired dimensions and can maintain the tube wall-thickness within remarkably small tolerances. It is interesting to note that 3 mm diameter tube is about 250 ft (75 m) long when fully drawn out. (Fig. 1.1.)

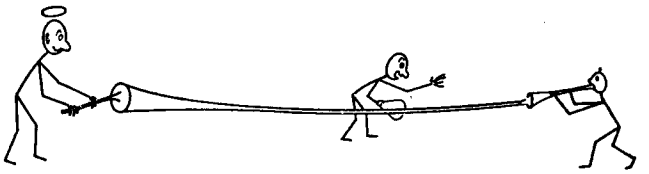

FIG. 1.1. Tube drawing by hand showing two members of the team walking backwards, drawing and blowing the hot glass into a tube, under the direction of a third member who is measuring the tube diameter.

The second method is a continuous machine method. A ribbon of molten glass flows from a furnace pot on to a hollow, tapered, rotating mandrel where it is further melted into a smooth, continuously rotating mass. This glass flows off the end of the mandrel and is drawn away by rollers and cut into lengths. The diameter and wall-thickness of the tube are maintained by controlling the temperature of the glass, the rate of drawing off, the air pressure blown through the hollow mandrel, and the speed of rotation of the mandrel. (Fig. 1.2.)

The third method is also a continuous machine method, and, except that the molten glass flows vertically through an annular orifice instead of on to a rotating mandrel, it is similar to the second method. (Fig. 1.3.)

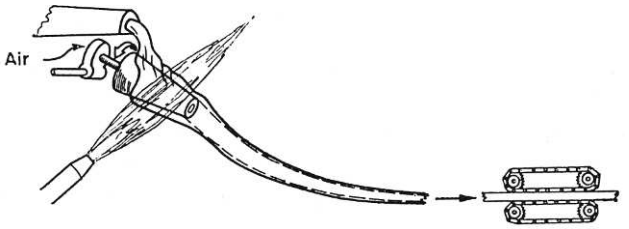

FIG. 1.2. Tube drawing by machine. The Danner Process. The molten glass from the continuous furnace flows on to the hollow rotating mandrel and is drawn off as a tube.

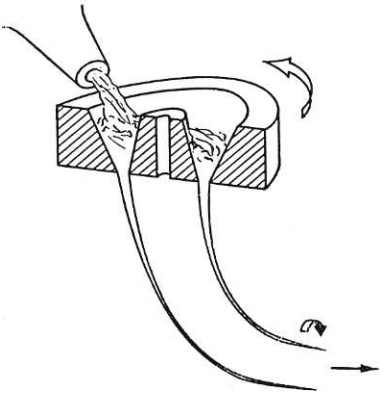

FIG. 1.3. Tube drawing by machine. The Vello Process. The molten glass flows into the rotating "sink", here shown in section, and emerges as a tube. The tube is drawn away by rollers or tracks similar to those in Fig. 1.2.

These brief descriptions will indicate that glassmaking and tube blowing are skilled and exacting industrial processes. A detailed treatment is given by Phillips[1] and by Threlfal.[2]

The glass used in laboratories represents a small fraction of the total weight of glass made in any year, which is said to be about the same as the annual production of steel.

The Composition of Glass

The number of possible glass mixtures is very large indeed; over 6500 have been made and the ingredients and properties recorded. These mixtures may contain, in various proportions, any of about half the elements in the periodic table. Each mixture results in a glass with physical, chemical and optical properties differing from those of all other glasses.

The simplest, and by far the most common type of glass made, is known as soda-lime glass. It contains approximately 73% silica, 13% calcium oxide and 14% of alkalis. It is used for window glass and for the vast majority of the many and varied bottles and containers made.

Glasses of this type contain an additive, known as a planing agent, which reduces the viscosity of the molten glass in the refractory pot, and so allows gas bubbles and fragments from the walls of the pot to float to the surface. Unfortunately, such planing agents tend to cause the glass to crystallize on subsequent reworking in the burner flame, and it is therefore unsuitable for laboratory glassblowing.

However, glassmaking firms have so perfected their manufacturing techniques that, even without planing agents, their products are remarkably free from gas bubbles, known as airlines, and from fragments of refractory material, known as "stone". Another defect, rarely seen in modern glass, is known as "cord" or "striae". Cord appears in glassware and tubing as a wavy line, the line being the boundary between regions of the glass with slightly different refractive indices and is the result of imperfect mixing of the ingredients. Airline, stone and cord have little, if any, effect on the working properties of glass, or on its mechanical strength, but they spoil its appearance and are undesirable.

Glasses intended for use in the glassblower's workshop are, for the most part, supplied in the form of tubing and rod. It is most important that successive consignments of any one kind of glass have the same physical, and to a lesser extent chemical, properties. This is important from the glassblower's

point of view since his materials must join together satisfactorily.

These glasses may be conveniently divided into three broad types: (1) soda glass; (2) borosilicate glass; (3) special glass.

Soda glass

Soda glass[3] is used for some neon-sign glassware, for many kinds of laboratory apparatus that are not subjected to high temperatures, are of fairly simple design and do not require glass of very heavy wall-thickness. Soda glass is readily worked and annealed in a coal-gas–compressed-air flame. It can be used to make matched glass-to-metal seals with chrome iron alloys. The maximum service temperature is 450°C and the thermal shock resistance is 115°C. The softening temperature of soda glass is about 560°C, beyond this temperature no further linear expansion is measurable, but a higher temperature is essential for working the glass.

The maximum service temperature for a glass is the highest temperature at which the glass can be used without introducing deformation or permanent strain.

The thermal shock resistance is a measure of the temperature range through which glass vessels, of normal wall-thickness, may be suddenly cooled without breakage.

The coefficient of thermal expansion of glass is a most important physical property. It is defined, for all solids, as the increase in length per unit length per degree rise in temperature. The coefficient of thermal expansion is a ratio and so has no units. It is, however, necessary to quote the temperature scale used.

The thermal expansion of soda glass, between 20°C and 350°C, is about $9{\cdot}6 \times 10^{-6}$ per °C.

New soda glass is very stable in the flame provided the working time is not prolonged or the flame temperature too low.

Let us diverge for a moment to consider a widely accepted

definition of glass. "Glass is an inorganic substance in a condition which is continuous with, and analogous to, the liquid state of that substance, but which, as a result of reversible change in viscosity during cooling, has attained so high a degree of viscosity as to be, for all practical purposes, rigid."

Glassmakers must select the proportions of the ingredients of their product so that, on cooling, no crystallization occurs. If, however, the surface of the glass is exposed for a considerable time to the solvent action of liquids, or to the moisture in the atmosphere, then part of the ingredients of the surface layer may be leached out. Thus the composition of this surface layer is changed and the chemical balance upset. The surface layer may then crystallize when the glass is heated and subsequently cooled.

This surface crystallization destroys the normal transparency of the glass and is therefore known as devitrification. It occurs in old glass and in glassware that has had prolonged use in the laboratory. It does not, however, become apparent until an attempt is made to repair or modify the glass in the flame. Such devitrified glass should be discarded.

Soda glass:	*typical composition* (%)
Silica (SiO_2)	70·5
Alumina (Al_2O_3)	2·6
Calcium oxide (CaO)	5·7
Magnesium oxide (MgO)	2·9
Sodium oxide (Na_2O)	16·3
Potassium oxide (K_2O)	1·2
Boric oxide (B_2O_3)	0·5
Sulphur trioxide (SO_3)	0·2

Borosilicate glass[4]

A number of borosilicate glasses suitable for general laboratory glassblowing have appeared during the last 65 years, and are now firmly established.

From the following comparisons it will be seen that the ad-

vantages obtained from using borosilicate glasses make it a more desirable material to use for laboratory purposes than soda glass. This is borne out by the fact that most full-time laboratory glassblowers prefer to use them, and most laboratory workers specify them for their glassware.

Advantages. They have a much lower coefficient of expansion and are therefore less liable to failure when suddenly heated or cooled. The apparatus is able to have a greater wall-thickness, and consequently greater mechanical strength, without affecting the thermal strength. They have a greater resistance to chemical attack and are less liable to surface deterioration with age; consequently devitrification is less likely to occur. Further, contained liquids and solutions are less liable to contamination by material leached from the glass wall. They are harder and more resistant to surface abrasion which lowers the mechanical strength.

If the glass is borosilicate many items of complex glassware, that would be impossible or very difficult to make from soda glass, can be fabricated, and repaired.

Disadvantages. Borosilicate glasses are more expensive than soda glasses.

They require an oxygen, or oxygen-enriched air supply to the burner or handlamp, since they have a high working temperature range. This temperature range, although higher, is also shorter than that of soda glasses, and the glass must be shaped and tooled quickly as it soon sets again when removed from the flame. Pin-holes are more likely to occur in borosilicate glass joins, and extra attention must be paid to cleanliness and to thorough melting of joins.

Borosilicate glasses have a maximum service temperature of the order of 500°C and, with some compositions, 600°C. Selected types are used to make matching seals with iron–nickel–cobalt alloy, with molybdenum and with tungsten. The softening temperature depends on the composition and is about 625°C. The thermal expansion also depends on the composition and varies from $3{\cdot}3 \times 10^{-6}$ per °C for ordinary borosilicate glass (widely used for laboratory glassware) to $7{\cdot}2 \times 10^{-6}$ per °C for

special types used in graded seals. The annealing temperature, depending on composition, is from 510°C to 600°C.

Borosilicate glass:	*typical composition* (%)
Silica (SiO_2)	80·80
Aluminium oxide (Al_2O_3)	2·20
Total Iron as Ferric oxide (Fe_2O_3)	0·04
Calcium oxide (CaO)	0·10
Magnesium oxide (MgO)	0·05
Sodium oxide (Na_2O)	4·20
Boric oxide (B_2O_3)	12·50
Chlorine (Cl)	0·10

Special glasses

Lead glass.[3] This is extensively used in the electric lamp and radio valve industries and for many electronic tubes. It seals readily to platinum and to copper-clad wires, giving a strong vacuum tight seal. Lead glass joins to soda glass. It is remarkably stable in the correct flame, showing no sign of devitrification. It must, however, be worked in an oxidizing flame, otherwise the lead oxide, near the glass surface, will reduce to metallic lead. This causes the surface to become blackened; it then loses its appearance, and will not join or work satisfactorily. It is not always possible to reoxidize this lead.

The thermal expansion coefficient is $9{\cdot}05 \times 10^{-6}$ per °C.

The maximum service temperature 350°C. The thermal shock resistance 120°C, and the annealing temperature 430°C.

Lead glass: typical composition (%)	
Silica (SiO_2)	56·0
Alumina (Al_2O_3)	1·3
Lead oxide (PbO)	30·0
Potassium oxide (K_2O)	8·0
Sodium oxide (Na_2O)	4·6

Silica.[5] When glassware must withstand operating temperatures higher than those recommended for borosilicate glasses, then a glass known as vitreous silica, or fused quartz and sometimes as quartz glass, is used. Pure, or 100%, silica SiO_2 is diffi-

cult to make and work as the silica tends to evaporate at the working temperature. The glass in common use is therefore about 99·8% SiO_2. The working temperature is in the region of 1800°C, and the glassblower must wear protective glasses. Chance Bros. "Protex", grade C, $\frac{1}{8}$ in. thick, are recommended. The expansion coefficient is $0{\cdot}5 \times 10^{-6}$ per °C and the annealing temperature is 1050°C. Flame annealing is adequate for laboratory silica ware of 2 mm wall-thickness. The plastic range of hot silica is relatively short, and graphite and molybdenum tools, rather than mouth blowing, are used for many shaping operations.

Silica tubing is available in a wide range of sizes, and in four types. The first type, known as Satin Surface Vitreosil, is translucent and has smooth interior and exterior surfaces. It is used to sheath thermocouples and to sample furnace and chimney gases. The second type has a rough exterior surface, is used in electric furnace construction and is known as Sand Surface Vitreosil. The third type results when Sand Surface tubing is subjected to additional fusion. It has smoother interior and exterior surfaces and is used for combustion and analytical operations at ordinary and reduced pressures. It is known as Glazed Vitreosil. The fourth type is highly transparent to visible light, to ultraviolet and infrared radiations. It is mechanically stronger and more resistant to devitrification than the translucent types and is recommended for high vacuum work. It is known as Standard Transparent Vitreosil Tubing. Transparent silica tubing is much more expensive than the translucent types. The manufacturer's reference handbook, *About Vitreosil*, and their descriptive leaflets, T1, T2 and T3, should be studied before ordering stocks of tubing and rod.[5]

A type of silica glass,[6] with physical and chemical properties very nearly the same as fused silica, is manufactured in America and obtainable in some other countries. It is known as 96% silica and its composition is as follows:

Silica SiO_2	96·5%
Boric oxide B_2O_3	3·0%
Aluminium oxide Al_2O_3	0·5%

The working temperature is about 1520°C and so it can be joined, blown and bent without risk of evaporating the silica. This glass can be melted with premix burners designed to use hydrogen, coal gas, or liquid petroleum (LP) gases together with oxygen.

The coefficient of expansion is $0{\cdot}8 \times 10^{-6}$ per °C, which is considerably less than that of borosilicate glasses and very little more than pure silica, so the annealing properties are good.

The maximum service temperature is given as 900°C.

Further information about this glass can be obtained from Corning Glass Works, Corning, New York.

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CHAPTER 2

Glass Tube and Rod

Storing Tube and Rod

Where the annual consumption of tube and rod is small, i.e. if no part-time or full-time glassblower is employed, the stock should be stored in its original cartons on easily accessible horizontal racks or shelves in the glassware store. The glass will then be protected from chemical fumes and vapours, from atmospheric dust and moisture, and it will be under the care of the storekeeper. When glass tubes are stored in such a way that their weight is not properly supported, they tend to bend under their own weight. This bending will become permanent with time. Although it does not make any noticeable difference when the tubing is used in relatively short lengths, badly bent tubes are unsuitable for many items of glassware, such as jacketed columns or very long condensers. The packing used by some glass-tube manufacturers is scientifically designed so that the individual lengths are supported in such a way that the bending moment induced in the glass, by its own weight, is at its minimum value.

Tubes stored in a vertical, or near vertical, position have their weight supported by one thin, fragile and often damaged end. Such ends are then liable to further damage. Short lengths become inaccessible, broken fragments scratch other tubes, weakening them and spoiling their appearance.

Tubes and rods should always be removed from the storage rack by lifting them clear of other tubes or rods. The glassblower who selects a tube from the middle of a tightly packed bundle then seizes the end with his fingers, drawing out the tube through the whole length of the bundle, is not only risking

deep cuts on his finger-tips, but is almost certainly scratching the outer surface of the selected tube and that of two or three others. Scratched tubes are spoiled in both appearance and strength.

If the demand for glassblowing is fairly regular, then a few lengths of the sizes in demand should be kept in the workshop, convenient to the glassblower's bench. This stock should also be stored in horizontal racks and protected from dust as far as possible.

Considerable care should be taken to keep glass of different kinds separated. It can be very annoying indeed to find, at a critical stage, that the tube being joined to an almost finished piece of glassware is of a quite unsuitable kind. There are several methods of deciding whether a tube is of lead, soda, borosilicate or silica. They are all, however, poor substitutes for efficient glass storage.

In general, only soda and borosilicate glass will be kept in considerable quantities. These can be stored in separate racks. A third rack can be reserved for special glass, which should be clearly labelled.

Dimension Tolerances in Tubes and Rods

All hand- and machine-drawn tubes and rods are subject to dimension variations. Glass rod is rarely perfectly straight, or perfectly circular in cross-section, nor is the mean diameter uniform along the length. Glass tubing is likewise rarely perfectly straight, and the inside diameter, the outside diameter and the wall-thickness vary round the circumference and along the length.

Such imperfections do not affect any but the most precise glassware. In most cases experimental and other errors are much greater than variations in tube dimensions. The magnitude of these variations will be understood if glassmakers' specifications are studied. Some indication of the tolerances to be expected will be found in the condensed Table 1.[1]

TABLE 1. MEDIUM WALL TUBING

Outside diameter (mm)	Tolerance (mm)	Wall-thickness (mm)	Tolerance (mm)
from 6 to 14	±0·5	1·5	±0·25
15 24	±0·5	1·75	±0·25
26 40	±1·0	2·0	±0·25
42 48	±1·0	2·5	+0·5 0·25
50 56	±1·0	2·75	±0·5
59 80	±1·5	3·5	±0·5
84 100	±2·0	4·0	±1·0

Capillary tube outside diameter will lie within ±1 mm of the specified size while the tolerance in the bore varies from ±0·25 mm for the 0·5 mm bore to ±0·5 for the 3 mm bore.

Rod of nominal diameter 3·5 mm will be accurate to ±0·5 mm and the largest size, 20 mm, will be accurate to ±1·0 mm.

The above figures refer to the best quality machine-drawn tubing and rod. Special glasses are often made in relatively small quantities; the tubing will then be hand drawn and the dimensional tolerances will be greater.

Precision Bore Tubing[2]

The accuracy of the dimensions of machine-drawn tubing is inadequate for such applications as high quality graduated glassware, syringe barrels, interchangeable manometers and many electronic devices which must have metal parts accurately located with respect to the glass-tube envelope.

To meet such needs precision bore tubing is available, such tubing is made from machine-drawn tubing of normal dimensional standards, but selected for freedom from airlines and stone.

The stock tube is mounted horizontally with a long mandrel running through the bore. The mandrel is accurately machined and polished for straightness, roundness and diameter, these being the critical factors in that they determine the finish and

degree of accuracy of the final product. The tube is locally heated by a small electric heater mounted over a horizontal track. As the heated glass reaches the correct viscosity, it is drawn out horizontally and its diameter decreases until the glass fits closely over the rotating mandrel. The air pressure inside the tube is considerably reduced by a vacuum pump, to assist with the drawing down process.

The bore sizes[3] produced by this reprocessing technique range from 1 mm $\pm$0·01 mm to 100 mm $\pm$0·04 mm.

Precision bore tubing is normally made from borosilicate glass, but is also obtainable in other glasses including silica.

Tubing and rod can also be obtained with the outside diameter ground to a specific size,[4] accurate to $\pm$0·0005 in. ($\pm$0·013 mm), and concentric with the axis.

Cross-sectional shapes other than circular are also obtainable, these include square, rectangular, triangular and D-sections.

All such tubes are expensive and so should be bought and used only if their special dimensions and form are essential to the function of the apparatus in hand.

Coloured Glasses

Coloured glass tubing and rod have a limited use in the laboratory glassblowing workshop. Most of them belong to the soda or lead glass types, and they will join readily to glasses of the same type. Coloured glass rod and transparent coloured glass tubing are not difficult to work. Those of the lead glass type must, of course, be heated in an oxidizing flame. Considerable skill and experience are required for working opaque or translucent glass tubing as the wall-thickness must be kept reasonably uniform and direct inspection is not possible.

Coloured glasses are used for decorative ware, as distinguishing marks on glass apparatus, as easily identifiable metal sealing glasses, and as light filters.

Glassmakers add certain inorganic substances to the usual glass ingredients before melting them in the refractory pot. In

addition to the desired colour, they must produce glass whose working properties and expansion coefficient are very nearly the same as those of the same type of clear glass.

Although quite small quantities of additive are sufficient to colour the glass, the final colour, at room temperature, depends not only on the quantity of the additive and on its purity, but also on the combination of additives used.

Table 2[5] shows the colours produced by various additives.

TABLE 2

Additive	Colour
Silver	Yellow
Copper oxide	Green and blue
Colloidal copper	Ruby red
Cadmium sulphide alone	Yellow
and with selenium	Shades of bright red and orange
Arsenic with lead oxide	White opal
Selenium	Pink, red, red–brown
Cerium dioxide and titanium	Yellow
Didymiums	Green (u.v. filters)
	Lilac red
Uranium	Fluorescent yellow–green
Sulphur	Amber
Sulphur with lead, iron, nickel or cobalt	Deep black
Colloidal gold (traces)	Ruby, brown, violet
Iron oxide	Blue, green and amber
Manganese oxide	Amber
Manganese oxide with iron oxide	Pink, deep purple, black
Chromium oxide	Green
Excess chromium oxide	Crystallizes out to give spangled or aventurine glass
Phosphoric acid	An ingredient of white opal glass or promotes transparency to u.v. or opacity to i.r. when used with ferrous iron
Nickel oxides and cobalt oxides	Brown, purple, deep blue

Ordering Stock

The quantity of the various sizes and types of glass tubing and rod ordered at any time should be enough to last for about 18 months. The existing stock should be used before the new stock. Buying stock calls for considerable experience and foresight, and some skill in making an informed guess as to the glassblowing workshop's future needs.

While it is always better to over-order rather than to under-order, it should be kept in mind that a comprehensive range of glass tube and rod sizes can cost at least a thousand pounds (£1000), and that unless all the sizes are used up in a reasonable time they will take up much valuable space and tie up money that could be used for other purposes.

In general, single standard packages of medium wall tubing, up to about 30 mm o.d. (outside diameter), can be bought with confidence. Larger sizes, and light or heavy walled tubing, should be bought only when required, or when there is a foreseeable need for them.

Some caution should be exercised in buying any tubing greater than 50 mm o.d. for a glassblowing workshop which is not equipped with a glassblowing lathe. Such large sizes can be handled satisfactorily only by a skilled and experienced glassblower.

Such a glassblower will be happy to look after his own glass stock. He will be best able to estimate the annual consumption and future needs, and most interested in keeping the stock clean, orderly and easily accessible.

Standard quantities, or packages, can be purchased directly from the makers or from the distributors. Small orders and quantities of less than a standard package will often receive better attention from laboratory furnishers than from the makers. The former suppliers, however, make substantial additional charges for glass tubing and rod kept in their stores. It is always worth while to inquire about costs and delivery dates before placing orders of any size with laboratory furnishers, and to compare them with similar estimates made by the tubing and rod makers or distributors.

Measuring Glass Tubing

In general, the glass-tube stock used in the workshop will be taken from the storage rack, where it will be so arranged that no difficulty will be experienced in selecting a tube within $\frac{1}{2}$ mm of the required size.

Should it be necessary to measure the outside diameter of any glass tube to some reasonable degree of accuracy then a vernier caliper should be used.

This instrument will be graduated in centimetres and millimetres. In its simplest form there will be a sliding scale, 9 mm long, divided into 10 equal parts. Thus the difference between a scale division and a vernier division is one-tenth of a scale division.

To measure the diameter of a tube the caliper jaws are adjusted so that they just touch the tube walls at diametrically opposite points.

The scale is read off in whole centimetres and millimetres,

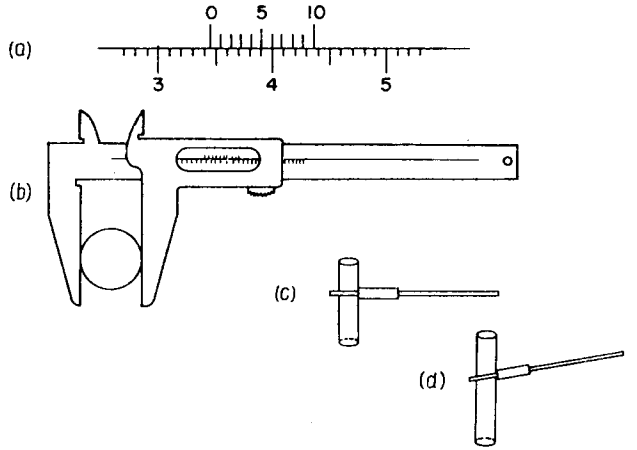

FIG. 2.1. (a) Vernier scale set at a reading 3·45 cm. (b) A typical Vernier caliper gauge. (c) Shows the correct angle for accurate measurement of tube diameters. (d) An incorrect angle, which will result in errors of measurement.

then the number of the vernier mark which most nearly coincides with a scale mark is noted.

In the vernier illustrated in Fig. 2.1, the reading is 3·4 cm, and the fifth vernier mark coincides with a scale mark. The fourth vernier mark is therefore 0·1 mm to the right of the nearest scale mark, and so the zero vernier mark is 0·5 mm to the right of the 4·0 mm scale mark. The diameter of the tube is therefore 3·45 cm.

The extended jaws of a vernier caliper gauge are used to measure the bore or inside diameter of tubes in the same way.

FIG. 2.2. Tapered gauge for measuring the bore of small diameter and capillary tubes. This gauge will give accurate readings when inserted in tubing with straight cut ends.

The bore of small diameter and capillary tubes can easily be measured with a taper gauge (Fig. 2.2).

Alternatively, the bore can be accurately measured by finding the largest number drill which can be inserted into the tube, and then consulting a table of number drill sizes.

Tube lengths should be measured off with a metre stick.

Every effort should be made to use and become familiar with the metric system. Since the dimensions on some sketches will be in inches and fractions of an inch, some care will be required to avoid confusing the measuring systems.

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3. Jencons Scientific Ltd., Mark Road, Hemel Hempstead, Hertfordshire, England. *Uniform High Precision Bore Glass Tubing*.
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CHAPTER 3

Laboratory Glassworking Hazards

SOME obvious hazards will be encountered in glassworking, namely, sharp glass edges, hot glass and tools, and the glassworking flame.

Hazards Due to Glass

Special care must be taken in the first few weeks of practical work to avoid injuries from these hazards. Handle glass-tube ends and all sharp-edged waste glass with great care. Thin, and therefore fragile, glass points are very dangerous, they easily penetrate the skin and can break off, leaving a small piece of glass embedded in the hands or fingers. Sharp-edged tube ends should not be placed in the mouth. These edges may cut the lips and bleeding from such cuts can be difficult to stop.

Experienced glassblowers often have a very untidy work bench, littered with broken tube ends and other waste glass. Such men have been glassblowing for some years and have acquired skill in handling dangerous glass fragments that looks deceptively careless.

All beginners should keep the bench-top free from waste glass and clear the bench after each assignment.

Burn Hazards

Burns to the hands are nearly always the result of absent-mindedness. It is advisable to allow heated glass time to cool before handling it and to acquire the habit of very lightly touching previously heated glass with the finger-tips first to ensure

that it is cool enough to hold. Burns are best avoided by giving undivided attention to the work being done and to nothing, and no one, else.

There is also some possibility of damage to clothing from hot glass. An overall or laboratory coat should be worn at all times, and hot glass waste should be placed on the bench top, well in from the front edge. Better still, it should be placed in a metal waste bin kept in a convenient place and for that purpose. No waste paper should be placed in this bin as it may be set alight by the hot glass waste.

It is not always possible to tell by inspection whether glass or tools are hot. The glassworking flame is always hot, so hands and sleeves must be kept out of harm's way. When making T-joins or bends it is necessary to develop a technique of holding and turning the glass in the left hand in such a manner that the hand does not pass through the flame.

All minor cuts and burns should be washed in an antiseptic solution and covered with an adhesive dressing. More serious injuries should be attended to by the first aid officer.[1]

Eye Hazards

Small glass fragments sometimes enter an eye. Should this occur, the eye should be covered with a soft pad. The injured person should be encouraged to keep his eye as motionless as possible and should be taken to the nearest hospital where a skilled doctor or nurse will remove the fragment. Such fragments will almost certainly have some sharp edges and much damage can be the result of movement of the eye, either by the injured person or by some unskilled efforts to remove the glass.

Prolonged exposure to heat, or infrared light, is said to cause cataract of the eye, and ultraviolet light emitted by hot borosilicate and silica glasses is said to cause corneal ulcers.

All hazards affecting the eyes can be very considerably reduced, and perhaps eliminated, if protective spectacles are habitually worn when doing any glassworking.

Sodium glass lenses give adequate protection from the rays emitted from hot soda glass. If any borosilicate glassblowing must be undertaken, then Didymium[2] lenses are recommended. Much darker lenses[3] are essential for eye protection if silica glass is worked in the flame.

Emphysema

All who do glassblowing are exposed to a small risk of developing a condition known as emphysema, a permanent inflation or over-expansion of the whole or part of either or both lungs. This condition reduces breathing efficiency and leads to shortness of breath on exertion, and, in extreme emphysema, shortness of breath when at rest. When emphysema has occurred it cannot be cured. It can, however, be avoided by taking simple precautions when blowing up molten glass.

Do not take a deep breath before blowing. Never blow with all the pressure you can muster; blow the glass into shape while it is plastic. A final hard puff when the glass has set will achieve nothing useful. When moulding ground joint cones and stopcock keys, use air pressure from the laboratory installation, controlled with a foot or manually operated valve.

Mercury

The presence of exposed mercury surfaces in any laboratory or glassblower's workshop must be regarded as a health hazard. Determined efforts should be made to clear up all spilled mercury, and to keep mercury containers covered.

Hazards Due to Repairable Glassware

Repairs to glassware have considerable hazards associated with them. The damaged apparatus may have contained inflammable substances or may have been washed with inflamm-

able solvents. These will evaporate and can easily form explosive mixtures with air, with consequent danger to the glassblower.

Poisonous substances may be left in or on glassware brought for repair and may find their way to the mouth or lips. The danger from radioactive materials is less obvious, but the results of accidentally ingesting such materials are indeed serious. Unless all glassware presented for modification or repair is clean and dry (the final rinse should always be distilled water and should be a thorough one) the glassblower is within his rights to refuse to undertake the work.

Glassware used for radioactive work should be brought to the glassblowing workshop by responsible senior staff only.

Hazards Due to Gas

Coal gas is poisonous and should not be inhaled, even in small quantities. The permanent gas pipes and taps rarely leak, but, none the less, they should be inspected at regular intervals and all defective fittings replaced. Modern rubber tube connections are often of poor quality and soon perish. Flexible plastic gas, air, and oxygen connecting tubes are much more reliable when properly fitted. They can be protected from excessive heat by enclosing them in flexible electrical conduit.

If Premix burners of any type are used, then, whether required by local law or not, a non-return valve of approved type should be fitted to the fuel gas line. Should compressed air or oxygen accidentally leak into the gas pipes, an explosive mixture may be formed, and the consequences can be serious indeed. A gas meter has been known to explode violently as a result of such a leak. Fortunately, no one was injured, but the risks are obvious.

When glassware, of even moderate capacity, is being worked in the lathe it sometimes happens that a few seconds elapse between turning on the gas supply to the burners and setting them all alight. Should the burner jets be directed towards open-ended tubes in the glassware, then an explosive mixture of gas and air may be formed within the apparatus, with disastrous results to

the glassblower's nerves and, sometimes, to the glassware in the lathe.

The remedy lies in always moving the burner jets away from open tube ends before turning on the gas supply and lighting up.

Glassware under vacuum may implode, and that under pressure may explode, because of shock waves set up by a door slamming or because of defects in glassware or supports; where such a possibility exists, the glassblower must be protected by covering large capacity bulbs with sacking or adhesive tape.

Cleaning Glass Tubing

Tubing from stock can be cleaned, inside and out, with a damp cloth. If very dirty it can be washed with hot water and soap powder, followed by thoroughly rinsing with tap water and a final rinse with distilled water. The tubes are then set up to drain.

Detergents help to speed up drainage but are themselves difficult to remove. They should not be added to the rinsing water for glass tubes intended for any type of gas discharge tube.

Glassware for repair or modification should be cleaned and dried by the user. He knows best what has been in the apparatus and what steps to take in cleaning it.

Chemical cleaning should be along the lines set out for sintered glassware by the makers of Pyrex crucibles and filters.

Fats and greases. Carbon tetrachloride or suitable organic solvent.

Albumen. Hydrochloric acid.

Organic substances. Warm concentrated sulphuric acid containing a little potassium nitrate and perchlorate. Immersion of the glassware in the former mixture for 12 hr or more is followed by thorough rinsing with water.

Cuprous oxide and iron stains. Hot concentrated hydrochloric acid with potassium chlorate.

Barium sulphate. Concentrated sulphuric acid at 100°C.

Mercury residues. Hot concentrated nitric acid.

Mercury sulphide. Hot aqua regia.

Silver chloride. Sodium hyposulphite.

Transparent Vitreosil apparatus must be given special care, as cleanliness is most important. At the working temperature many materials combine with silica and cause stains or devitrification. In addition to normal cleaning the surfaces are wiped with cotton wool dipped in alcohol. The cleaned surfaces must not be handled as finger marks are sufficient to cause devitrification.

Hydrofluoric acid, in concentrations of 10–40%, is often recommended for cleaning heavily contaminated glass surfaces. This acid can cause serious and painful burns. Reliable watertight rubber gloves must be worn and the hands should be coated with anti-acid barrier cream. The gloves should be carefully rinsed in tap water before taking them off.

Blowing

Although the art of fabricating glassware from tubing involves blowing, with the mouth or using air pressure from some other source, to shape or reshape the molten glass, it is generally considered that such blowing plays a minor, though essential, part in the work. In general, the glassblower acquires skill in blowing from experience.

However, when a technician undertakes a course in laboratory glassblowing, he will have but limited time to acquire extensive experience and must rely on a rather poor substitute, instruction.

The magnitude of the excess internal pressure required to blow out molten glass depends on three factors:

(1) The wall-thickness of the glass.
(2) The temperature of the molten glass, and therefore its surface tension and viscosity.
(3) The internal diameter of the heated tube or bulb.

Thick-walled glass requires rather greater excess internal pressure to blow it up than thin-walled glass. It is therefore important that the molten glass be of nearly uniform wall-thickness. Small

variations in wall-thickness can be removed by first blowing gently to shape the thin sections, which cool first, then increasing the air pressure to blow up the thicker glass, which cools more slowly and will remain plastic for a longer time.

Since the viscosity increases rapidly as the glass cools, it is clearly necessary to have the glass really hot if subsequent work will involve blowing. It is inconvenient to measure the temperature of the heated glass, so there is no substitute for experience in estimating when the glass is hot enough for the operation envisaged.

It can easily be shown that the excess pressure inside a spherical liquid bubble of negligible wall-thickness of surface tension λ and radius r is P (see Fig. 3.1), where $P = 4\lambda/r$.

The excess internal pressure required to expand this bubble will be P_1 where $P_1 > P$. Since λ for any type of glass will depend on the temperature only, then at any given temperature

P is inversely proportional to r

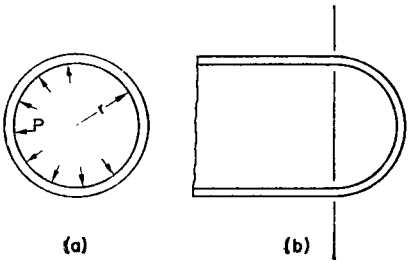

FIG. 3.1. The excess internal pressure required to blow out a test-tube end (b), is the same as that required to blow out a spherical liquid bubble (a).

and if r is very small, e.g. the bore of a thermometer tube or the bore of capillary tubes of less than 1 mm, then P_1 will be large and may be greater than can be produced by mouth blowing.

It is recommended that, where small bore capillary tube has to be melted and blown, a rubber policeman or stoppered rubber tube be attached to the open end of the capillary glass and

squeezed to blow up a bulb on the heated closed end. This bulb can then be blown out by mouth pressure and prepared for further work (see Fig. 3.2).

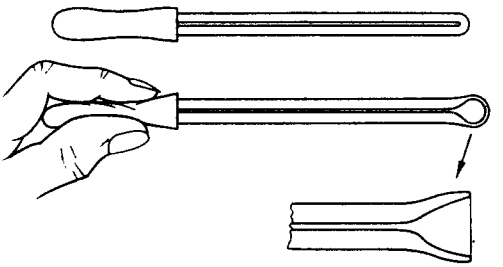

FIG. 3.2. Blowing out a capillary tube end with the aid of a small rubber bulb or a stoppered rubber tube.

Since most of the glassware made at the bench has a relatively small internal volume, the quantity of air that must be blown into it to appreciably raise the internal pressure is small. The technique used to blow up the molten glass in such glassware is the same as that employed by many wind-instrument players. The lips are closed and the mouth inflated by the lungs so that the cheeks are slightly expanded. The throat is then closed so that normal breathing through the nose can be resumed while the cheeks remain expanded and can exert a controlled and variable air pressure in a tube held in the lips. This technique should be practised at every opportunity until it can be applied without conscious effort. It is doubtful if the physical action of closing the throat, to isolate the mouth from the other air passages, can be described or even demonstrated. Most glassblowers will be happy to demonstrate the result: closed lips, slightly inflated cheeks, accompanied by normal breathing through the nose.

All open tube ends on a piece of glassware, except the one used for blowing, should be securely stoppered with cork or asbestos stoppers or rubber plugs. Small leaks must be avoided, as they upset blowing techniques and excessive loss of air can be harmful to the chest and diaphragm muscles.

While it is possible to mould stoppers and cones for interchangeable ground glass joints and for hollow stopcock keys by mouth blowing, this is an exhausting and quite unnecessary task

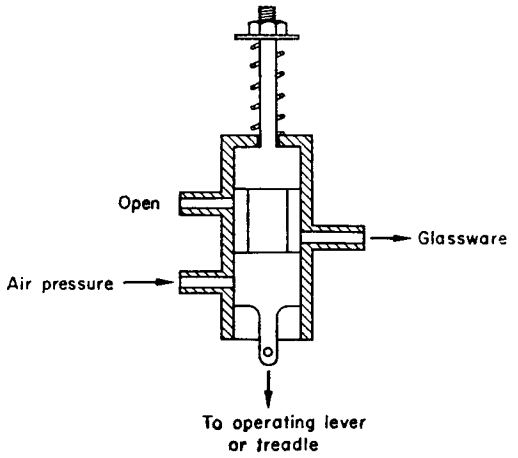

FIG. 3.3. A two-way valve for controlling air pressure. The air in the glass being heated can expand to atmosphere when the piston is in the position shown. Air pressure is admitted to the molten glass when the lever is depressed.

if air from a compressor is available. A simple two-way valve made in the mechanical workshop from brass or aluminium, connected up as shown in Fig. 3.3, and operated by hand or by foot-treadle, will do a better job with much less effort.

References

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3. The Thermal Syndicate, P.O. Box No. 6, Wallsend, Northumberland, England. 1965 Catalogue.

CHAPTER 4

The Workshop

WHERE possible a room should be set aside for glassblowing only. In such a room the glassblower will be able to keep his equipment, stock and accessories ready to hand. He will also be protected from the disturbing noises of hammering, sawing and drilling that are normal in a laboratory workshop, and will enjoy much-needed privacy when he tries out a new technique or makes up some complicated or unusual apparatus.

Location

The location of the workshop depends on a number of considerations. It should be reasonably central with respect to the science departments; with some bias in favour of the chemists who will, no doubt, make most frequent demands on its facilities. The workshop should be near the bulk store where most of the tube stock will be kept; and close to the unloading bay so that heavy gas or oxygen cylinders can be easily replaced. A ground-floor workshop has many advantages. At best there must be some compromise in choosing a workshop site: a separate building would be ideal; at worst a corner of a laboratory may have to serve.

Lighting[1]

The temperatures of flames and the working condition of hot glass are very difficult to estimate if the illumination level in the workshop is too high. An air–gas or oxygen–gas flame cannot

be seen in full sunlight. Subdued daylight from the northern sky in the northern hemisphere, and from the southern sky in the southern hemisphere, is best. Should the sun shine through the windows at any time of the day, Holland blinds or Venetian blinds can be fitted. If the day-time illumination is too high for most of the year, the window glass can be painted on the inside with a pale green translucent paint.

Artificial lighting over the glassblowing bench and machinery can be provided by fluorescent tubes which reduce contrast and shadows. Additional lighting may be required where drawings and sketches are made, and for reading vernier calipers or a micrometer.

Floor

The floor area of the workshop will depend on the amount and nature of the work to be undertaken. Not less than 40 m^2 (400 ft^2) will be necessary if no limit is to be put on the glass-blowing service required. The floor bearers should be strong enough to support a glassworking lathe and an annealing oven, if such equipment is to be installed.

A hardwood floor surface has much to recommend it. It is easy to walk on and has a pleasing appearance. Temporary and permanent fixtures are readily secured to wood. However, this type of surface is seldom waterproof. Should a flood occur, and floods of some size are almost inevitable in laboratories or workshops which use water, the ceiling of the room below will suffer. Spilled mercury droplets tend to lodge in the cracks and joins; such droplets are difficult to pick up and will cause a health hazard if left on the floor. Good quality linoleum is satisfactory when correctly laid and cemented down; it is readily damaged by hot glass fragments and by broken glass trodden underfoot.

Bare concrete floors are unsuitable since they are tiring to walk and stand on, and increase the damage done to any glass tube or apparatus that may be accidentally dropped.

Ceiling Height

A ceiling height of not less than 3 m (10 ft) is desirable so that the hot gases and the products of combustion from the burner flames may be readily dispersed. Should the ceiling height be less than 3 m, or if the room temperature is uncomfortably high, then a metal fume hood should be fitted above the burners to remove the hot gases.

Ventilation

Efficient draught-free ventilation should be provided, especially in hot climates. Artificial heating is necessary in cold weather. Ideally the glassblower's workshop would be air-conditioned so that temperature and humidity are under control.

Acoustics

Prolonged exposure to the noise of burner flames and workshop machinery can cause industrial deafness, or partial loss of hearing for some frequencies. Industrial deafness is incurable and where workshop noise makes conversation difficult every effort, including consultation with an acoustics expert, should be made to reduce noise.

The Bench

A glassblower's workbench is an obvious necessity. It should be about 0·75 m (30 in.) high, from 0·75 m (30 in.) to 0·90 m (36 in.) wide and of substantial construction. The top should be of wood and about 1·5 m (5 ft) long to allow freedom of movement in handling full-length tubes. The working area on the bench top may be covered with asbestos board (preferably the soft variety) to protect the surface from accidental burns. Apart from the

small fire hazard, such burns soon make the bench top unsightly. If the asbestos cover and that area of the wall behind the burner are painted flat black or dark green, the flame and the hot glass will be more easily seen, and eye-strain reduced.

A drawer, or a set of drawers, situated within easy reach of the seated position, is useful for holding small tools, cork stoppers and other accessories.

The Seat

While some glassblowers prefer to stand at the bench it is considered good practice to sit when working. Again there are many who prefer a comfortable chair with a padded seat and back rest. In general, a laboratory stool is all that is necessary. In the seated position the elbows should rest comfortably on the bench top, just in from the edge. Both feet should be on the floor. An easy elbow position is essential. Should this need a stool of such height that one's feet do not reach the floor they may be rested side by side on a stool leg brace.

Many hours will be spent seated at the bench, so it is important that the working position be free from discomfort. The body-weight must be evenly distributed, the feet should not dangle in mid-air, nor should the legs be crossed or stretched out under the bench in a relaxed attitude. Try a number of stools of different heights to find one that is suitable, and as a last resort alter the seat height.

Other Furniture

If a steady demand for a wide range of glassware is anticipated a considerable area of extra bench space will be desirable. The assembly of elaborate vacuum systems and other extended glass apparatus must proceed, in part at least, in the workshop. Space will also be required for a large mercury tray so that this mercurial metal can be confined to a reasonable area. A high vacuum system and a baking oven will be essential if vacuum jacketed

apparatus is required and they will occupy much bench space. Glassware awaiting repair or modification, partly completed and finished work all take up bench space.

A number of drawers and cupboards fitted under the benches are useful for storing stopcocks and ground-glass joints; for asbestos paper, tape and string; for special tools, burners and handlamps; and for all the vast conglomeration of odds and ends that accumulate over the years.

Sink Bench

Inevitably long glass tubes and glassware will have to be washed; the workshop should have a large sink with hot and cold water and a lead or rubber covered draining board about 1·7 m (5 ft 6 in.) in length. (This will not be necessary if such facilities are available in a nearby wash-up room.)

Tools

Cutting tools

A single-cut hand file, 15 cm (6 in.) long will cut soda or borosilicate tubes of less than 30 mm diameter. It should be kept in good condition; when difficulty is experienced in making a deep file-mark on the tube wall the file must be resharpened on a grindstone as follows: the narrow faces of the file are ground off so that four new edges are exposed. Very little metal need be removed but care should be taken to avoid overheating the file and altering the temper of the metal. (See Fig. 4.1.)

Cast-steel glass knives are obtainable. They have wooden handles and are more convenient than files for cutting small diameter or thin walled tubes (Fig. 4.2). Cast-steel knives must be sharpened frequently, especially if they are used to cut borosilicate glass. A No. 120 carborundum stone is used to grind a well supported edge, of the form shown in Fig. 4.2.

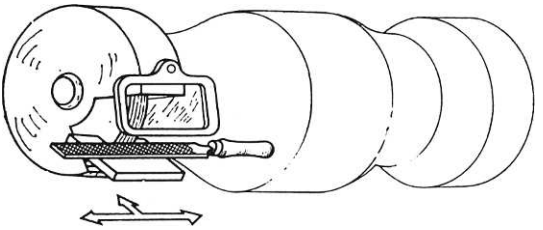

FIG. 4.1. Renewing the edges on a glass-cutting file. A small thickness of metal is ground off the narrow faces with an engineer's grindstone.

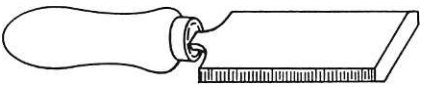

FIG. 4.2. A cast-steel glass-cutting knife.

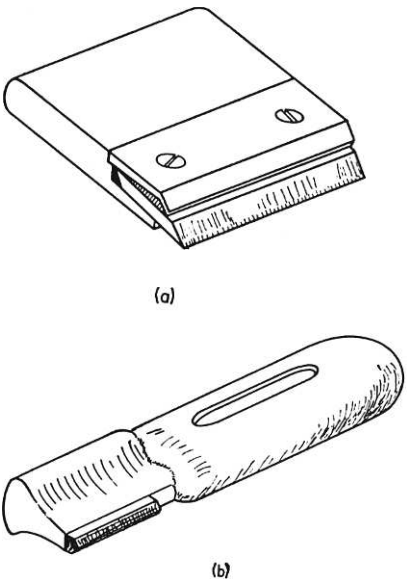

FIG. 4.3. Tungsten carbide glass-cutting knives.

Tungsten carbide glass-knives (Fig. 4.3) are much more expensive, but require less frequent sharpening and are more reliable than files or cast-steel knives; again, the edge should be well supported. A special stone must be used: Mason Master Green Grit C80MV Grinding Wheel is recommended. A useful glass-cutting knife can be made by grinding a suitable edge on a piece of high speed hack-saw blade, and attaching it to a wooden handle as shown in Fig. 4.4.

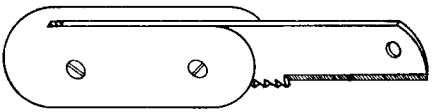

FIG. 4.4. Glass-cutting knife made from a high-speed hack-saw blade.

A set of heavy gauge iron wire hooks are convenient for cutting off short ends and large diameter tubes. The hook should fit the tube and a number of sizes will be required. (See Fig. 4.5.)

An electrically heated nichrome wire of heavy gauge is a convenient substitute for the wire hooks (Fig. 4.6). It can be obtained complete with transformer, switch and wire supports, or can be made in the electrician's workshop. The hot wire easily adjusts itself to any tube diameter within its range of sizes.

Cutting wheels of various kinds can be used if very large numbers of short glass tubes of the same size are required. Thin

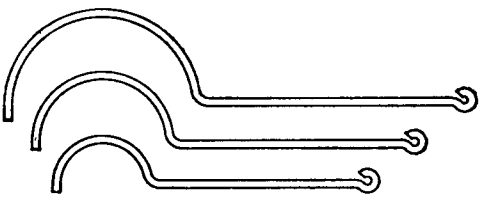

FIG. 4.5. Iron wire hooks. The glass tube is scratched with a knife or file, the wire hook is heated to cherry red. The tube is held horizontally and rotated with the scratch in contact with the hot wire. A crack will immediately appear and travel round the tube, leaving a clean straight cut end.

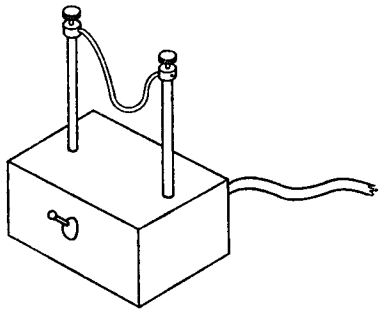

FIG. 4.6. Electrically heated wire. The hot wire is used to cut straight ends in the same way as the iron wire hooks. The wire easily adjusts itself to fit any tube diameter.

hardened steel discs, 30 cm diameter and rotating at a peripheral speed of 1200 m/min will readily cut lead and soda tube. The glass is pressed against the edge of the disc and slowly rotated. The disc makes a deep scratch and generates enough heat by friction to start a crack. The glass is easily broken off with a straight end.

Such discs are unsuitable for low-expansion silica tubes.

A thin brass disc with a diamond impregnated edge, rotating at a periphal speed of 900 m/min, can be used to cut through heavy walled tubing and rod of all sizes. An adjustable flow of cooling liquid is necessary. Such discs are used in the optical glass workshop but have few applications in the glassblower's workshop.

Sharp-edged carborundum wheels, rotating at 1500 m/min, are used in lamp and radio-tube factories for cutting up lead and soda tubing of up to 18 mm diameter, in the same manner as the hardened steel discs.

The best power-driven tool for cutting glass tubing has a thin rubber bonded carborundum disc 0·3 m (12 in.) diameter and 0·16 cm ($\frac{1}{16}$ in.) thick, rotating at a peripheral speed of 1500 m/min (5000 ft/min). This disc will cut all types of glass tubing and rod. A good flow of water is required. The tube or rod is pressed against the cutting edge and the cut gradually

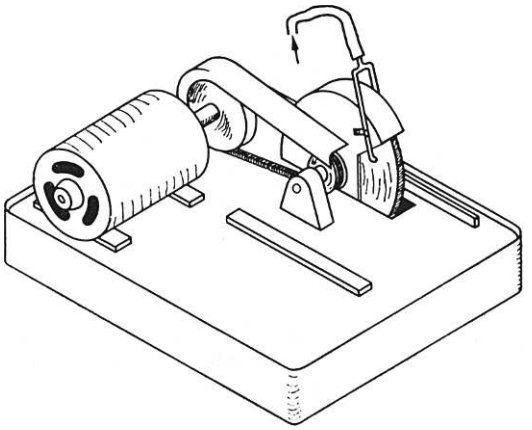

FIG. 4.7. Glass-cutting machine fitted with thin rubber-bonded carborundum disc. Water cooled. The previously marked tube is pressed gently against the cutting edge and rotated until the cut penetrates the glass wall all round.

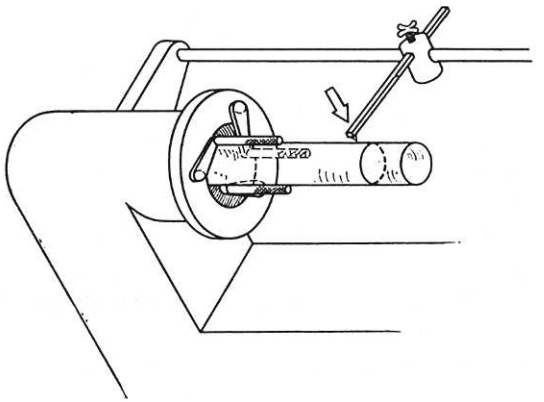

FIG. 4.8. A diamond-tipped tool, used to cut glass tubing held in the lathe chuck. The lathe chuck is turned by hand; some care is desirable to avoid pressing too hard on the tool bar and to avoid extending the scratch beyond one complete turn.

deepened all round. It is helpful to make a straight circumferential pencil mark round the tube, or to guide the disc with a heavy rubber ring stretched over the tube and adjusted until it presents a circular face in a plane at right angles to the tube centre line. There is a small risk of the bonding medium contaminating the glass edges. Such contamination is easily ground off on the side of the disc (Fig. 4.7).

Correctly mounted diamonds are convenient for cutting glass tube into uniform short lengths. A variety of tools, fitted with diamond edges, are available from laboratory furnishers. Glazier's diamonds can be adapted to suit individual requirements, e.g. for cutting large diameter borosilicate tubes in the lathe. The diamond must be mounted by an expert, otherwise the edge will be quickly blunted, or the scratch will be unsatisfactory. The simplest type is shown in Fig. 4.8.

Other bench tools

A set of brass flanging and reaming tools[2],[3] can be made in the metalwork class. A basic set should consist of those shown in Fig. 4.9. The blades are made from 18 gauge brass sheet, finished with smoothly rounded edges and brazed to 3 mm ($\frac{1}{8}$ in.) brass rods 7 cm (3 in.) long. The handles are of hardwood or some heat-resisting material. Brass is the preferred material for the blades since it is a relatively soft metal and will not scratch the glass. The blades are kept lightly coated with beeswax or Nuboid to provide lubrication. They should not be overheated in the burner flame, or they will melt.

A pair of medium weight forceps, 15 cm (6 in.) long, are useful for pulling off excess glass.

Tools of this type are too small and light for lathe work; a set of carbon rods, from 6 mm ($\frac{1}{4}$ in.) to 25 mm (1 in.) in diameter mounted in wooden or brass tube handles, is required. Carbon paddles can be bought or made to suit personal requirements. They are useful for smoothing joins, large flat flanges and flat bottomed vessels made in the lathe. (See Fig. 4.10.)

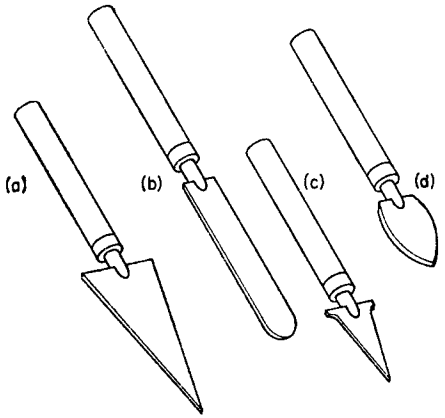

FIG. 4.9. Hand tools with brass blades. Used for bench work: (a) is used to make moderately large flanges; (b) is used to make rims on tube ends; (c) is used to rim and flange small diameter tubes; (d) is a general purpose flanging and rimming tool.

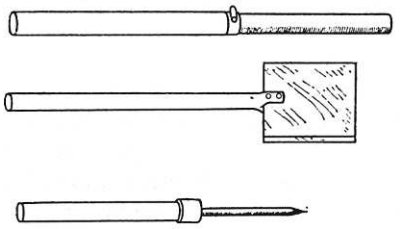

FIG. 4.10. Hand tools for lathe work. Made from carbon and fitted with brass tube handles.

FIG. 4.11. A rubber blow-tube with saliva trap.

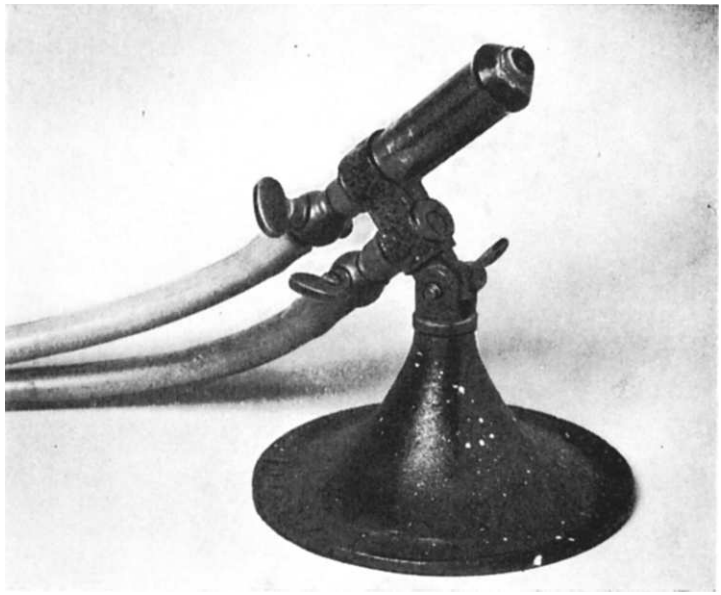

PLATE 4.1. An orifice-mixing bench burner with interchangeable air jets.

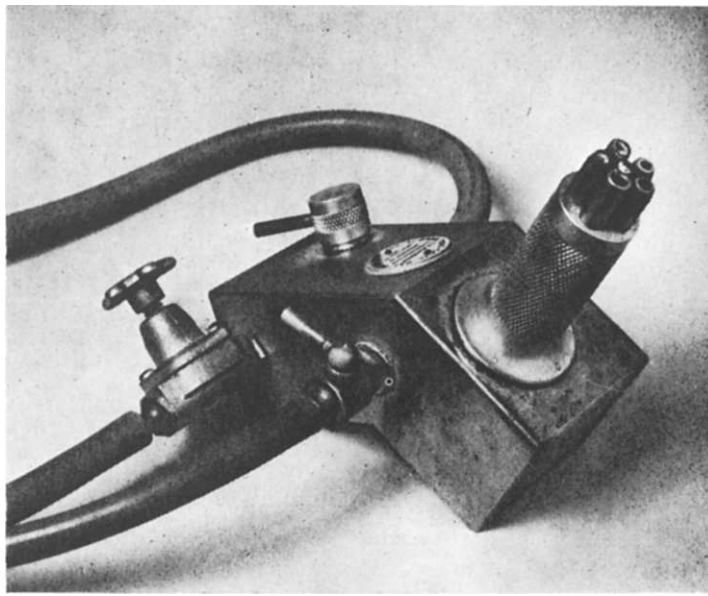

PLATE 4.2. A premix bench burner with turret head and provision for attaching a handlamp. Made by L. V. D. Scorah, 44 Northfield Road, Birmingham 30, England.

Steel forceps, 25 cm (10 in.), with heat-insulated handles should be included in the basic tools for lathe work.

About 2 m (6·5 ft) of 6 mm ($\frac{1}{4}$ in.) bore rubber tubing fitted with a glass mouthpiece is essential for blowing up glass worked in the lathe or by handlamp. It can also be used to blow into glassware worked in the bench burner if it is inconvenient or impossible to blow directly through an open end. (Fig. 4.11.)

A metal swivel, made from brass or aluminium will eliminate any twisting of the rubber tube. A number of patterns are offered by laboratory furnishers. Two such swivels should be obtained as some jobs must be blown from both ends simultaneously (Fig. 4.12).

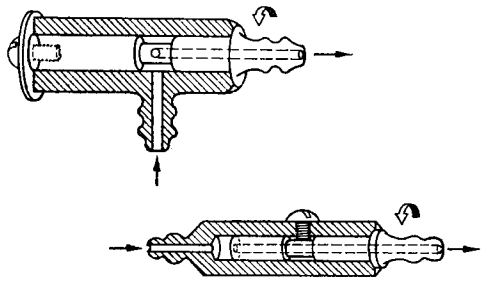

FIG. 4.12. Blowing swivels. These swivels are necessary when glass must be blown in the lathe. They are also used to blow into glass worked at the bench burner if there is no convenient end for mouth blowing.

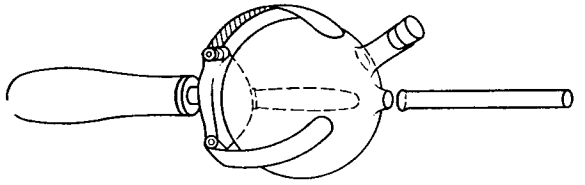

FIG. 4.13. An adjustable bulb holder.

Flask holders enable flasks, beakers and glassware of large diameter and relatively short length to be held and rotated in the flame so that joints and tubes may be added. A set of these holders is desirable in the absence of a glassworking lathe. One is shown in Fig. 4.13.

A wooden stand, with a large V-notch, supports long lengths of tubing. The more sophisticated glassblower's donkeys serve the same purpose, and can also be used in pairs to support heavy tubes when they are being joined or flanged. Wooden racks are used to support cooling glass, and can be made to a number of designs; some of which are shown in Fig. 4.14.

Short lengths of tubing are easily cut off with the aid of a heavy gauge metal plate, secured to a substantial base and with a deep, sharp edged V-notch cut in the top. A large retort stand fitted with two clamps will support glass tubes to be joined with a handlamp. (Fig. 4.15.)

A generous supply of cork and asbestos stoppers is essential; one or two of each size should be fitted with a glass tube handle, about 20 cm long and 8 mm in diameter.

A number of short lengths of rubber tube, of different diameters, and with one end plugged with a cork stopper, should be prepared. They are used to close tube ends, and are easily mislaid.

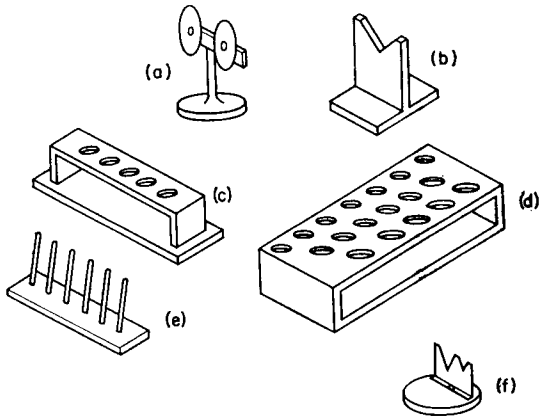

FIG. 4.14. Bench-top accessories: (a) and (b) are used to support long lengths of tubing; (c), (d) and (e) are typical cooling racks made from wood; (f) is a useful cutting aid. Small diameter tubes or capillary tubes, or rods are marked with the knife and placed in a notch so that the mark is directly above the edges; a sharp blow with the back of the knife will cause the glass to break off cleanly at the mark.

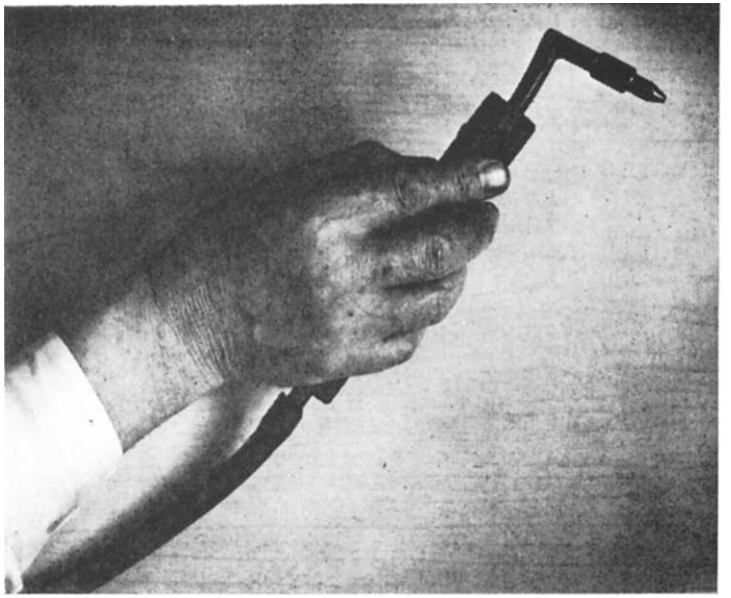

PLATE 4.3. A simple handlamp suitable for use with Scorah bench burner.

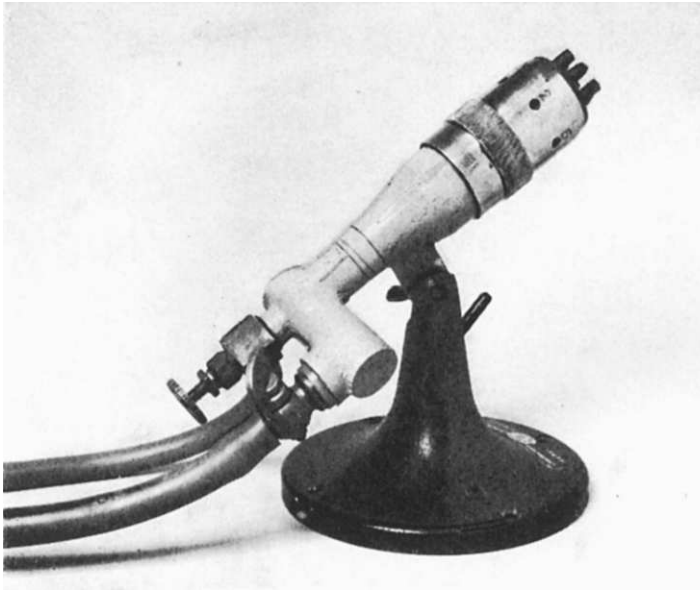

PLATE 4.4. A compact premix bench burner, also suitable for lathe-work. Made by Jencons, Scientific Ltd., Mark Road, Hertfordshire, England.

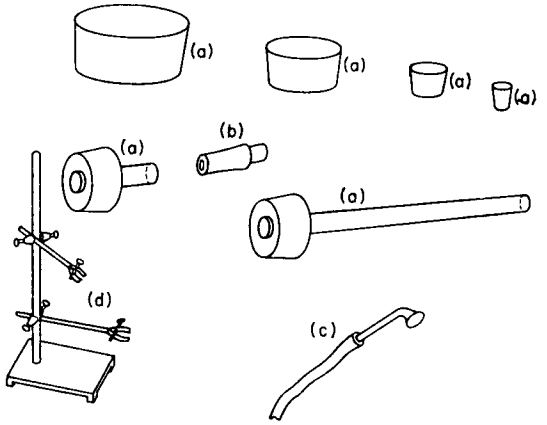

FIG. 4.15. Bench-top accessories: (a) cork stoppers with and without blowing tubes and handles; (b) rubber tube plug, or policeman; (c) rubber blow-tube with plain mouthpiece; (d) laboratory stand with two clamps.

BURNERS AND HANDLAMPS

The tool used to produce flames suitable for working glass tubes is variously named; torch, burner, blowpipe, lamp, cannon, fire and gun. In the interests of simplicity that used for bench work will be referred to as a bench burner or a burner; that used in the hand will be called a handlamp or a lamp. The multiple flame type of burner is used in some workshops for bench work and is universally used for working glass in a glass-blower's lathe; they will be referred to as crossfires and are shown in Plate 4.5.

There are two types of burners, embracing bench burners, handlamps and crossfires.

The first type is shown diagramatically in Fig. 4.16. Compressed air, or oxygen-enriched air, at about 0·34 kg/cm^2 (5 lb/in^2) flows through the central tube and emerges from the small bore jet. Coal gas (town gas or manufactured gas) at about 28 cm (11 in.) water gauge flows through the annular space and

emerges from the burner port. The oxidant and gas diffuse into one another and their proportions can be controlled with stopcocks. The resulting flame depends on the proportion of these and can be varied from a large brush to a small sharp flame.

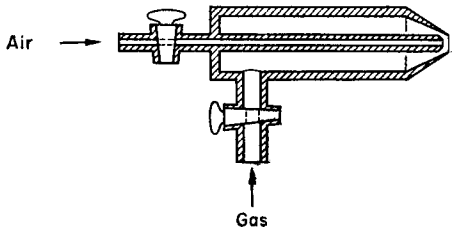

FIG. 4.16. An orifice-mixing bench burner shown in section.

This type of burner is usually supplied with a number of interchangeable jets which give a wide range of flame sizes (Plate 4.1).

Multibore jets are also used; they give a hotter and more flexible flame.

Since the mixing takes place outside the burner barrel there can be no flashback if the proportions of fuel gas and oxidant are misjudged.

The second type is known as a premix burner. The air (or oxygen enriched air or oxygen) is fed to the burner at a pressure of about 1 kg/cm^2 (15 lb/in^2) and passes through a small bore jet. The high velocity oxidant entrains fuel gas and the mixture burns with a hot and relatively quiet flame (Fig. 4.17). The size of the flame depends on the bore of the burner port; such burners are usually supplied with a number of interchangeable ports or flame units (Plate 4.2).

Premix burners are also made with one or more rows of small orifices. This type is known as a ribbon burner and is used to

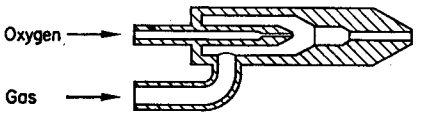

FIG. 4.17. A premix burner shown in section.

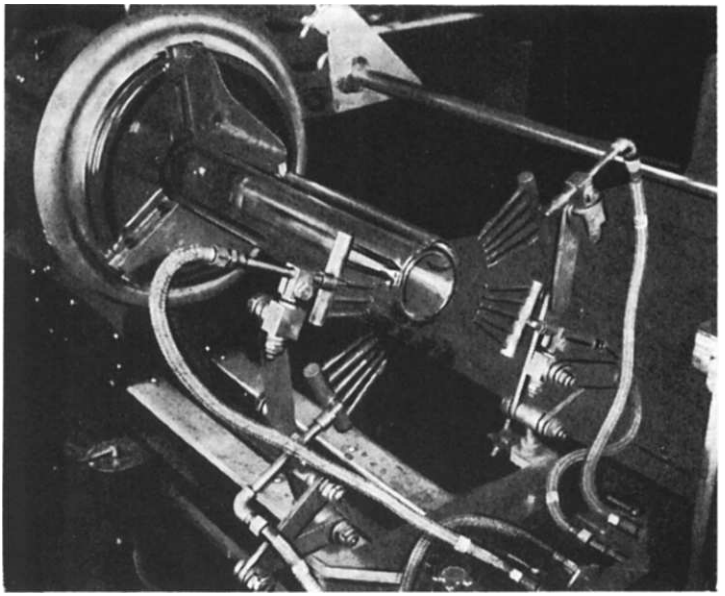

PLATE 4.5. Premix crossfires with interchangeable jets. Supplied by Heathway Engineering Co. Ltd., Hillingdon, Middlesex, England.

heat long lengths of tubing so that they may be bent into smooth curves.

When the small orifices are grouped in concentric circles the resulting flame is very hot and stable.

The flow of fuel and oxidant mixture through a premix burner orifice must be kept above a minimum rate, depending on the fuel, to avoid flashback. Flashback occurs when the mixture inside the burner barrel ignites with an explosion, which extinguishes the flame.

There are a number of burners, lamps and crossfires available. Most of them are designed to burn coal gas–air, coal gas–air–oxygen or coal gas–oxygen mixtures. Such burners are not suitable for use with hydrogen, natural gas or liquid petroleum gases.

Orifice-mixing burners are the cheapest and are adequate for working soda glass. Premix burners should be used for borosilicate glass. They give a hot flame when used with oxygen and are relatively quiet. However, orifice-mixing burners may also be used for borosilicate glass, but the oxygen enriched air gives a very noisy flame which is undesirable in the vicinity of lecture-theatres or classrooms.

The following bench burners, handlamps, ribbon burners and crossfires have been used by the author and have proved satisfactory.

For working lead and soda glass using coal gas and compressed air:

Stone-Chance Limited. Bornkessel Dual Purpose Bench Burner 250/12/5 × 9.

Stone-Chance Limited. Bornkessel Ribbon Burner 754 (Premix).

Stone-Chance Limited. Bornkessel Hand Torch 26/1501.

Stone-Chance Mark II Flamemaster Hand Torch (Fig. 4.18).

For working borosilicate glass:

A variety of orifice-mixing bench burners and handlamps using oxygen enriched air and coal gas.

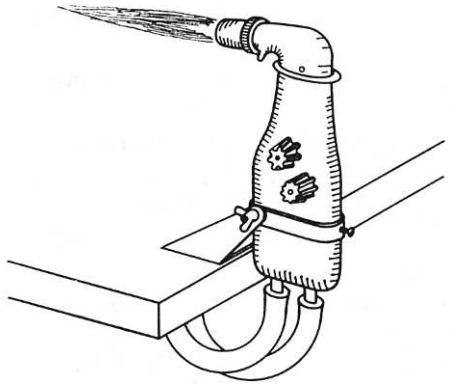

FIG. 4.18. A Flamemaster handlamp with bench clamp.

Scorah oxy-coal gas bench burner (Premix) (Plate 4.3).
Jencon's Rota jet bench burner (Premix) (Plate 4.4).
Stone-Chance Mark II Flamemaster Hand Torch (Premix).
Crossfires supplied by Messrs. Heathway, Lathe Manufacturers.

For working silica:

Stone-Chance Mark II Flamemaster Hand Torch (Premix) with oxy-hydrogen. An oxy-coal gas handlamp (Premix) made in the workshop. Crossfires by Heathway with oxy-hydrogen.

The Thermal Syndicate Ltd., makers of Vitreosil, recommend the British Oxygen Co.'s No. 109616 lead welding torch for working tube up to 38 mm diameter, and the same company's No. 35069 lead welding torch for very small silica work.

These torches burn oxy-hydrogen or oxy-propane. Oxy-coal gas may be used if the gas pressure is not less than 15·25 cm water gauge.

"Prince" sheet metal welding torch supplied by New Zealand Industrial Gases. Used with oxy-hydrogen.

Fuel Gases

Coal gas

Coal gas is obtained when coal is heated to about 1000°C in the absence of air. Its composition depends on the coal from which it is obtained, an average is:

Hydrogen 50%; methane 32%; carbon monoxide 8%; nitrogen 6%; ethylene 4%.

Coal gas, also known as town gas and as manufactured gas, is the most suitable fuel gas for working lead, soda and borosilicate glasses. It can be used with burners and handlamps of simple design to give a wide range of flame sizes and intensities.

The gas pressure, as measured on a simple U-tube manometer, is usually about 20 cm (8 in.) water gauge.

The reticulation of the gas to the benches should be through permanent piping installed by a competent gas fitter. The principal gas-pipe should be of not less than 19 mm ($\frac{3}{4}$ in.) bore. It should be fitted with an isolating stopcock of similar bore so that modifications or repairs may be carried out without cutting off the supply to other users on the same line from the meter.

A non-return valve should also be fitted to prevent oxygen or compressed air being admitted to the gas line and creating an explosion hazard. This is a simple flap valve which closes when the flow of gas is reversed. It has a large area and opens readily with little or no impediment to gas flow in the right direction. Non-return valves of approved design are normally obtainable for the gas supply company, who, in many cases, insist on installing them when oxygen or compressed air is to be used.

Branch pipes, leading from the main pipe to the bench stopcocks, should be of short length and not less than 12·5 mm ($\frac{1}{2}$ in.) bore. The stopcocks should be of similar bore. The type of stopcock fitted to laboratory benches, and used to control the gas flow to a bunsen burner, is too small for glassblowing work. Small-bore gas-pipes, stopcocks (or gas taps) and connecting tubes will considerably reduce the flow of gas to the burners, and,

notwithstanding an adequate static pressure of 20 cm water gauge, the kinetic pressure at the burner may fall to 5 cm or less and the flame size will be insufficient for bulb blowing, tube bending, or annealing.

Very old gas-pipe installations may appear to be of adequate bore, yet they may be so encrusted with internal deposits that the effective bore is much reduced. Such installations should be replaced.

If the pressure at the gas-meter is too low to meet all the demands made on the supply it may be necessary to install a gas booster. This is simply a pump which raises the pressure above that provided by the gas works. The installation of a gas booster can seriously affect the gas supply to other users on the same main pipeline, and raise the pressure in adjoining laboratories to undesirable levels, it is therefore essential to consult the local gasworks engineer before such an expensive programme is undertaken.

Hydrogen

Commercial grade hydrogen can be used for glassblowing if there is no coal gas available. Hydrogen is much more expensive and its use involves the hire and replacement of heavy cylinders. A fuel–gas pressure regulator (Fig. 4.19) is also necessary, since the cylinder pressure is, when full, 140 kg/cm^2 (2000 lb/in^2), and the line pressure need be no more than 20 cm water gauge.

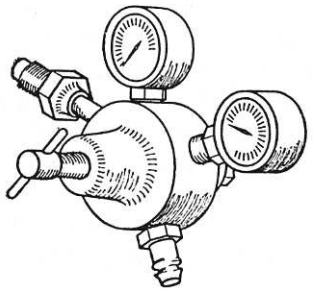

Fig. 4.19. A pressure regulator with two gauges.

Air–hydrogen flames from an orifice-mixing burner may be used for working soda glass; extra care must be taken to preheat the glass, and since the flame is at a higher temperature than a similar air–coal gas flame the glass melts very quickly and becomes rather difficult to control. This type of flame may also be used for working borosilicate glass if no other burner is available.

Oxy-hydrogen flames, as produced by a premix burner with a 1 mm bore flame unit, can be used for borosilicate glass but are not recommended. They are widely used for working silica and are satisfactory if the work is carefully planned and prepared so that the melting time is kept to a minimum. Prolonged heating causes the silica to evaporate and condense on the colder regions of the tubing. This condensation is in the form of an opaque white deposit which spoils the appearance of the article and is difficult to remove.

The pressure in any fuel–gas cylinder should not be allowed to fall to zero on the regulator gauge; the cylinder should be refilled while the positive pressure inside is sufficient to prevent any air or oxygen entering the cylinder and mixing with the fuel.

Natural gas

Natural gas, also known in its crude form, as marsh gas and fire-damp, consists mainly of methane (CH_4). Other gases are present in various proportions, the most important being ethane, propane and butane. Natural gas is an important fuel since it contains about 95% combustible hydrocarbons. This gas is used in many glassblowers' workshops in America. Since it is relatively cheap and efficient, its future utilization in other countries is probable.

Liquid petroleum fuels[4]

Propane (C_3H_8) and butane (C_4H_{10}) are both gaseous at atmospheric pressure but can be liquified at pressures of 7 to 10 kg/cm^2 (100–140 lb/in^2). They can be stored and transported

as liquids provided they are kept at an appropriate pressure. LP (liquid petroleum) fuels can be used for glassblowing. Special burners must be employed, and the makers of the gas should be consulted before equipment is purchased and installed.

The LP fuels are sold under various trade names and consist of liquids containing various proportions of propane and butane, with small amounts of other hydrocarbons, which vaporize when released at atmospheric pressure.

They are denser than air and although they readily disperse when released to the air, LP fuels may accumulate in closed spaces, such as cupboards, and can then be an explosion hazard.

They are normally odourless, and so have an odorizing agent added to draw attention to leaks.

They are tasteless, colourless and non-poisonous; none the less, because of the fire hazard, great care must be taken to ensure that all fittings and connections are gas-tight.

Properties of fuel gases

Air requirement is the minimum amount of dry air required for complete combustion, usually expressed as a volume/volume ratio.

Calorific value is the heat given out by complete combustion of a unit volume of the gas.

Specific gravity is usually given relative to dry air.

Burning velocity, or flame speed or flame propagation rate, is defined, in simple terms, as the speed of a flame relative to the gas–oxidant mixture. The burning velocity is of critical importance in flame unit design.

The burning velocity of a gas depends on a number of variable factors, such as the ratio of oxidant to fuel gas, the amount of non-combustible material in the gas and of non-oxidizing material in the oxidant. It also depends on the conditions under which burning takes place; whether vertically up or down, or horizontally; and on the diameter of the containing vessel. It is known that for each gas–oxidant mixture there is a minimum

TABLE 3. PROPERTIES OF FUEL GASES SUITABLE FOR GLASSWORKING[5]

Fuel	Air requirement (vol./vol.)	Relative burning velocity	Relative cost per unit volume	Relative calorific value	Max. temperature (°C)		Specific gravity relative to dry air
					Air	Oxygen	
Hydrogen	2·4	9·2	11	290	2045	2660	0·07
Coal gas	4·4	2·2	1	480	1950	2730	0·5
Natural gas	9·5	1·0	—	960	1875	2930	0·55
LP gas	24–31	1·0	4	2520	1930	2760	1·5–2·0

diameter of tube through which flame will propagate. This principle is used in the Davy safety lamp: the flame of this lamp is enclosed in a fine-mesh wire screen which prevents inflammable gases outside the lamp from being ignited by the flame inside.

It can be assumed that a flame unit, or burner jet, will produce a serviceable flame if the burning velocity of the fuel–oxidant mixture is approximately the same as the velocity of the mixture at the burner. Should the burning velocity be too high the flame will flash back, i.e. the mixture within the flame unit will ignite. Should the burning velocity be too low the flame will blow off, i.e. the mixture will not ignite at the burner port but will burn at some distance from it. Such flames are unsatisfactory.

The art of designing flame units for fuels with relatively low flame propagation rates lies in surrounding the main flame port with a suitable number of pilot flames of appropriate diameter, through which the fuel–oxidant mixture emerges at the required velocity. An arrangement of pilot flames is shown in Fig. 4.20.

While it is possible to make flame units for air–coal gas, air–oxygen–coal gas and oxygen–coal–gas burners with the aid of normal workshop materials and tools, it should be noted that considerable experience and skill are required to make such flame units for other gases. In general it is recommended that the special burners and handlamps necessary for those fuel gases

be purchased. Much time and material can be taken up with experimental flame units and it is doubtful if the accumulated experience can have any real utility.

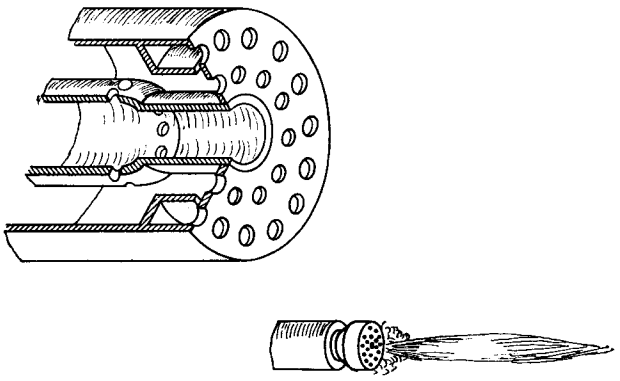

FIG. 4.20. Premix burner port provided with pilot flames, for gases with low burning velocity.

COMPRESSED AIR

Most laboratories have a compressed air plant installed in the building. The compressed air is then reticulated to all laboratories and workshops. The pressure at the glassblower's bench should be about 0·7 kg/cm^2 (10 lb/in^2), and where necessary this should be controlled by a pressure regulator.

Should it be necessary to purchase a compressor for the glassblower's workshop, one of adequate output should be obtained. The maker's specifications should be studied and, if possible, the selected type should be seen in operation before a final decision is made. Much inconvenience and frustration can follow the purchase of an unreliable or inadequate air compressor. As a temporary measure, and last resort, one may use foot-operated bellows. However, this type of compressed air supply demands that the attention of the glassblower be divided between maintaining a steady flow of air and rotating and manipulating molten glass. The latter tasks are usually quite enough for beginners.

Oxygen

A supply of oxygen is essential if borosilicate or silica glass-work is to be undertaken. Oxygen can be obtained in heavy metal cylinders or "bottles" of various capacities. The oxygen in a newly filled bottle is at about 140 kg/cm^2 (2000 lb/in^2) and the capacity is quoted in terms of *l* (ft^3) at atmospheric pressure. This ranges from 1130 *l* (40 ft^3) to 7000 *l* (240 ft^3). The largest size is heavy and should be moved from place to place with the aid of a cylinder trolley; it is otherwise convenient since replacements will only be required about every 14 days, depending on the type of work in hand. Oxygen bottles can be hired from the nearest supplier of industrial gases. The hire charge varies from place to place and the cost of oxygen is quite low, about 0·2 cents/litre.

The pressure in the oxygen pipeline need only be about 1 kg/cm^2 and a pressure regulator must be fitted to the bottle. A number of regulators of different types are available. The most convenient type is fitted with two gauges, one to indicate the pressure in the bottle and the other to indicate the pressure in the oxygen pipeline.

An instruction leaflet is supplied with each new regulator. This must be read and understood before the regulator is fitted to the bottle. Failure to carry out the instructions, particularly those covering the operations involved in shutting off the oxygen supply, can result in serious damage to the regulator seals, and the delay involved in effecting repairs or providing replacements. The flow of oxygen to any burner or handlamp is more critical then the flow of compressed air. This flow can be controlled by suitable needle or diaphragm valves and these are recommended for all oxygen control points.

Flames

When the type of fuel gas has been decided on, and the air supply compressor purchased and installed, it is advisable to become familiar with the burner and its operation. Check tube

connections to ensure that they are correct. Always turn on and light up the gas first, then turn on the air slowly. All changes in gas or air flow should be made slowly at first—there is a possibility that the flame will be blown out by excess air.

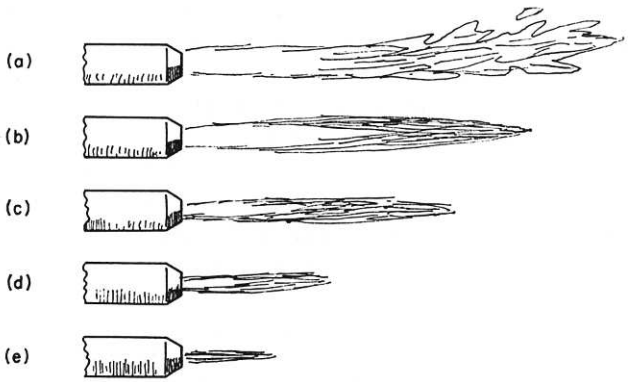

FIG. 4.21. Burner flames. (a) A gas flame with no air, used for preliminary heating and for annealing soda glass. (b) A large flame with very little compressed air flowing, for bending large radius curves in tubing and for annealing. (c) This flame has rather more air flowing than (b) and has a distinct blue inner cone; it is used for joining tubes of about 15–20 mm diameter. (d) Similar to (c) but smaller, suitable for joining tubes of less than 15 mm diameter. (e) A small sharp flame for joining rod and for local heating.

There is no substitute for experience in selecting the best size and intensity of flame for any given job. The aim should always be to heat just enough glass to carry out the required operation. The glass must always be rotated in the flame so that it is just beyond the blue inner cone. This is the hottest region in the flame; the temperature within the blue cone is very much lower. Typical flames are shown in Fig. 4.21.

FLAME QUALITY

Reducing flame. Should the oxidant supplied to a flame be insufficient to completely oxidize the fuel gas, oxygen will be taken from any available source. The blackening of lead glass in a reducing flame is the most appropriate example.

Neutral flame. When the oxidant and fuel supplied to the flame are such that combustion is complete and neither is in excess, the flame is said to be neutral.

Oxidizing flames. These are produced when there is an excess of oxygen. Such flames are essential for working lead glass. They are smaller and perhaps hotter than neutral flames and are used for most local heating.

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CHAPTER 5

Annealing

GLASS, like most substances, expands when heated and contracts as it cools. This can be demonstrated by supporting both ends of a 150 cm length of tube in V-shaped tube rests or in retort stands and clamps, and gently heating the tube along its length and on the lower part only. The heating can be done with a bunsen burner or a handlamp, and some care must be exercised to avoid heating the glass to softening temperature. The tube will be seen to bend downwards because of the increase in length of the lower part. When the heating is stopped the tube will cool and gradually recover its original shape. If heating is confined to the upper part the tube will bend upwards.

Should this expansion or contraction be prevented by any means, very powerful forces will be set up within the glass. Unless these forces are removed they may, and all too often will, break the glass.

Let us consider some definitions and their application to glass; some aspects of transient or temporary stresses and of permanent stresses.

The coefficient of linear thermal expansion. If a piece of straight glass rod of any diameter and of length L cm is heated uniformly through Δt°C, it will expand by some amount l cm. We can now say that the increase in length/unit length/°C rise in temperature is α, and

$$\alpha = l/L\,\Delta t/°\mathrm{C}.$$

Now if α is known for any particular type of glass we can calculate the increase in length of any given original length if we measure the rise in temperature.

Increase in length $= l = \alpha L \Delta t$.

α is therefore a multiplier and is known as the coefficient of linear thermal expansion of that type of glass.

In general, α is very nearly the same over the range between room temperature and that temperature above which the expansion cannot be measured because the glass has lost its rigidity.

Stress

When a rod of uniform cross-section supports a weight, the rod will be subjected to tensile stress if the weight hangs from the rod; and to compressive stress if the weight rests on the rod, as shown in Fig. 5.1.

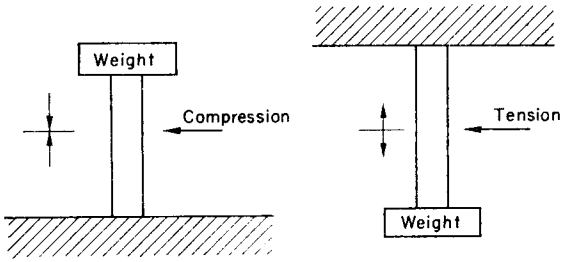

FIG. 5.1. The stresses introduced into a loaded rod.

Let the weight or load be W kg and the cross-sectional area of the rod be A cm^2; then the stress intensity σ will be

$$\sigma = W/A \text{ kg/cm}^2.$$

Strain

Under this load the rod will stretch or contract slightly. This change in length/unit length is known as strain. The tensile strain and compressive strain, or shortening, have the same value given by

$$\text{Strain} = l/L \quad \text{(no units)}.$$

Young's Modulus

For any given load W there will be some stress σ and a corresponding tensile or compressive strain. Provided the load is not great enough to break the glass the ratio of these two values will be a constant. This connection between stress and strain is known as Hooke's law, and the constant ratio as Young's modulus:

$$\text{Young's modulus} = \text{stress/strain}$$

and has the value $6{\cdot}2 \times 10^8$ g wt/cm^2 for glass.

EXAMPLE 1. Consider a glass rod, 1 cm long and 1 cm^2 in cross-sectional area, cooled through 1 °C, and prevented by some means from contracting. Take $\alpha = 9{\cdot}7 \times 10^{-6}$/°C.

The change in length if free to contract is

$$\begin{aligned} l &= L\alpha\Delta t \\ &= 1 \times 9{\cdot}7 \times 10^{-6} \times 1 \text{ cm} \\ &= 9{\cdot}7 \times 10^{-6} \text{ cm}. \end{aligned}$$

If the rod is prevented from contracting the tensile strain is equal to this change in length,

i.e. $$\text{strain} = 9{\cdot}7 \times 10^{-6}.$$

Since Young's modulus = stress/strain,

therefore $$\text{stress} = \text{Young's modulus} \times \text{strain}.$$

Taking Young's modulus for glass as $6{\cdot}2 \times 10^8$ g/cm^2,

$$\begin{aligned} \text{stress} &= 6{\cdot}2 \times 10^8 \times 9{\cdot}7 \times 10^{-6} \text{ g/cm}^2 \\ &= 6{\cdot}0 \text{ kg/cm}^2. \end{aligned}$$

EXAMPLE 2. Consider a glass rod with cross-sectional area 1 cm^2 and length 10 cm prevented from changing in length. Suppose that 1 cm only of the length is cooled through 1 °C and that the values of α and Young's modulus are the same as those in example 1.

The decrease in length $= l = L\alpha\Delta t$

$= 1 \times 9{\cdot}7 \times 10^{-6} \times 1$ cm
$= 9{\cdot}7 \times 10^{-6}$ cm;

again the strain $= l/L$
$= 9{\cdot}7 \times 10^{-7}$ cm

and the stress $= 6{\cdot}2 \times 10^{8} \times 9{\cdot}7 \times 10^{-7}$ g/cm^2
$= 0{\cdot}6$ kg/cm^2.

From these examples it will be clear that the stress in a cooled glass rod, prevented from contracting, depends on the magnitude of the fraction of the total length cooled and on the amount of cooling.

Let us now consider the effects of heating glass by taking two hypothetical examples.

EXAMPLE 3. Assume that a very thin flat sheet of glass can be heated at a central spot and that the flame does not spread over the sheet. On heating, a small circular area of glass will tend to expand and so to occupy a slightly larger area. The cool surrounding glass will now be subjected to tension across a section such as *ab* in Fig. 5.2. It will also be subjected to compression across a section *cd*. When the temperature of the central spot reaches that at which the glass just begins to soften the tension and compression will disappear. If the heated glass is now allowed to cool it will contract and tend to occupy a slightly

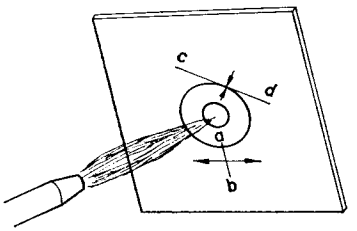

FIG. 5.2. Local heat applied to a thin sheet of glass *(inner circle)* will set up stresses in the surrounding glass *(outer circle)*. These stresses will be compressive at a tangent to the outer circle and tensile in the radial direction.

smaller area. This contraction will set up compression across *ab* and tension across *cd*. As we have seen these forces will be large.

It has been established that new glass will probably break if tensile stresses of about 420 kg/cm^2 are applied to it. Glass is much stronger in compression and, in general, 4200 kg/cm^2 are necessary before it fails. When the glass sheet has cooled to room temperature these stresses will persist and the glass, if not broken, will be deformed and no longer flat. This type of deformity is known as thermal strain.

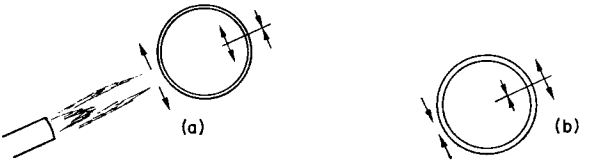

FIG. 5.3. Shows the effect of applying local heat to a thin slice of glass tube. At a radial section opposite the heated spot, compressive stresses are set up in the outer layer of glass, and tensile stresses are set up in the inner layer, as shown in (a). These stresses disappear when the heated spot begins to soften. When the glass is allowed to cool the heated spot contracts and sets up compressive stresses in the inner layer and tensile stresses in the outer layer as shown in (b).

Should the glass be allowed to cool before the softening temperature is reached all the stresses will disappear. The strain set up during such heating and cooling is known as transient thermal strain, as it is clearly temporary.

Transient thermal strain is also capable of breaking glassware, e.g. heavy walled soda glass milk bottles will break if quickly filled with boiling water.

EXAMPLE 4. Assume that a thin slice of glass, cut from a tube, is locally heated by a small flame, as shown in Fig. 5.3. The heated section of the ring will expand, tending to open out the ring. This will result in compression stresses being set up in the outer layers of the glass and tensile stresses being set up in the inner layers. As heating proceeds the temperature will reach softening point and, as before, all stresses will vanish. On cool-

ing to room temperature these stresses will be reversed, i.e. compressive stress will remain in the inside layers and tensile stress in the outer layers.

The foregoing examples are hypothetical; the glass is assumed to have two dimensions and the pattern of stresses is simple. All glass has, necessarily, three dimensions, and stress patterns can be very complex. In practice the glass conducts heat and the region surrounding the hot zone will not remain cool. Also, gas flames, however small, do spread over surfaces and, again, the heat cannot be confined to rigidly defined zones.

It follows from these arguments that any piece of glass will be permanently strained if part of the material is heated to some temperature at which the glass molecules are free to move relative to one another. Should this strain result in stresses above the breaking stress then the material will fail.

Such failure is in the form of cracks which have a characteristic shape. They usually circumscribe the heated zone and rarely pass through it. They usually extend through thin glass walls. With heavy walled glass, i.e. more than 2 mm, local heating of short duration can result in flakes of glass separating from the surface.

Failure of the material in completed glassware is obviously undesirable and can be avoided by a process known as annealing. There are two methods in common use in all workshops where glass is melted and shaped. The first, known as flame-annealing, involves heating all the glass surrounding and including the worked zone in a large bushy flame until a sodium yellow colour can be seen on the glass surface. The article is then set aside to cool. This method is only effective with fairly simple glassware provided the wall-thickness nowhere exceeds 2 mm.

The second method is more effective and is indeed essential with heavy walled or complex glassware. It is known as oven-annealing. A suitable oven is one which will heat up to about 600 °C in 1·5 hr, and, when the heating is discontinued, will cool to room temperature in about 10 hr. Annealing schedules vary for different glasses and also for different wall-thicknesses. Such schedules are made available by the glass manufacturers. An

oven operating as described above is suitable for laboratory glassware.

It should be noted that heating to some temperature below the recommended annealing temperature does not, even partially, reduce thermal strain.

The Thermal Strength of Glasses

It should now be clear that the magnitude of both residual (or permanent) and transient thermal stresses depends on the coefficient of expansion of the glass under consideration. This coefficient varies for different glasses from $0{\cdot}5 \times 10^{-6}$ per °C to nearly 15×10^{-6} per °C. The corresponding breaking stresses for new glasses varies very little from 420 kg/cm^2. It follows that the glass with the lowest coefficient of expansion will have the greatest thermal strength and will consequently require less careful annealing. Even if an oven is available it is, however, necessary to flame-anneal all complex glassware, for otherwise it may not survive till the oven is next used.

Experienced glassblowers flame-anneal all their products immediately after the work is completed. Indeed, they regard flame annealing as an essential part of making the glassware. Flame-annealing does not eliminate residual thermal stresses; it moves them to regions where the glass is a simple cylinder and spreads them over a length of this cylinder where they are less likely to cause breakdown.

Oven-annealing is a refinement, a step that makes doubly sure that residual thermal stresses are so reduced that they will not cause the glass to fail.

For some purposes glass is deliberately left under permanent stress. For example: pinch seals made for electric lamps are stronger if they are but partly annealed. One type of automobile windscreen is stressed to increase its mechanical strength. The method is as follows: the glass sheet is cut to size and all the edges ground and polished to a semicircular cross-section. The prepared sheet is bent to the required shape. The windscreen is now heated to some temperature above the annealing tempera-

ture but not so high that the glass deforms under its own weight. The entire surface of the glass is rapidly cooled by blasts of air. The glass below the surface layer cools more slowly, since it is a poor conductor of heat and this differential cooling sets up considerable compression stresses in the surface and tension stresses in the interior of the glass sheet. The process is controlled so that the residual internal stresses are too low to break the glass.

Externally applied mechanical stresses will break the windscreen when they are sufficiently great that they not only neutralize the considerable residual compression stresses in the surface layers, but also create tension stresses sufficient to break annealed glass.

Such prestressed glass cannot be cut, drilled or worked in any way, as damage to the surface layer would upset the balance of the internal forces and cause the glass to break.

Prestressed windscreens not only withstand large external forces but possess the great advantage, over other types, of breaking up into small and relatively harmless fragments should the car be involved in an accident.

Prince Rupert's drops provide an interesting and easily made example of the effects of stressing glass.

The end of a 5 mm soda glass rod, selected for freedom from airlines, is melted in an oxy-gas flame. When the end has gathered back and is about 6 mm in diameter, the flame is directed on to the rod just behind the hot blob, at the same time the rod is tilted up, almost vertically. A drop of molten glass will fall away from the rod but remain attached to it by a long tail. The hot glass drop is allowed to fall into a 500 ml beaker of cold water. The long tail is melted off so that about 3 cm of it is left attached to the drop, as shown in Fig. 5.4.

Provided that the drop is not too big and has therefore broken spontaneously in the beaker, and provided that the glass was free from airlines which would expand and relieve the internal stresses, then all attempts to break the Prince Rupert's drop will fail. If, however, the tail is broken off, the drop breaks, with an appreciable shock, into very small pieces.

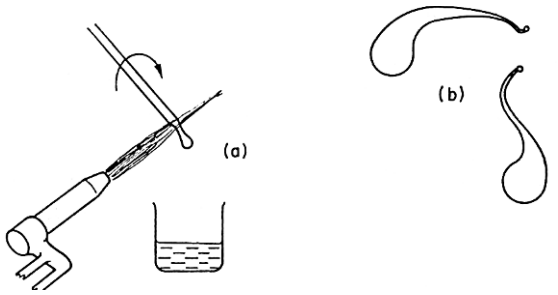

FIG. 5.4. Prince Rupert's drops. (a) Shows the heated glass about to drop into a beaker of water. Typical shapes for these drops are shown in (b).

Bolonga phials have disappeared from laboratory furnishers' catalogues. They were made of heavy walled soda glass and had a plain neck and a round bottom, as shown in Fig. 5.5. The outer surface of the phial was cooled quickly during manufacture and it would withstand heavy blows from a hammer. When a small

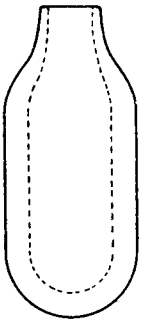

FIG. 5.5. A Bolonga phial.

piece of sharp-edged material, such as flint or steel, was dropped inside, the slight damage to the inner surface layer upset the balance of the internal stresses in the glass and the bottom of the phial would break off.

Strainviewer

The presence of residual thermal strain in glass that has been flame annealed cannot be detected with the unaided eye. The location of some of the glassware in an oven can be such that it may not reach the annealing temperature indicated by the temperature sensing device. Should failures occur in glassware that can only be due to excessive thermal stresses—and therefore strains—the annealing treatment must be inadequate, and some means of examining the glassware for strain should be used.

It is known that light transmitted by strain free glass obeys the laws of refraction; and that a ray of light transmitted by strained glass is split into two rays—the ordinary ray and the extraordinary ray. These two rays follow slightly different paths through the glass, they are plane polarized and their vibrations are at right angles to one another. This property of strained glass is possessed by other materials such as calcite crystals, and is known as double refraction or birefringence. If a beam of polarized light is passed through a piece of glassware and the emergent light is viewed through an analyser, those regions of the glass that are strained will show a brighter light intensity.

Two sheets of polaroid, a material which polarizes light passing through it, can be borrowed from the Physics Department and set up as in Fig. 5.6, to make a rough strain viewer capable

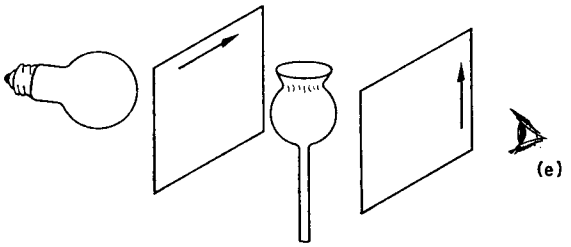

FIG. 5.6. A simple strainviewer showing the arrangement of the light bulb, the two sheets of polaroid with their planes of vibration at right angles to one another. The transmitted light as seen by an eye at *e* is then at its minimum intensity. The strain in any piece of glassware held between the polaroid sheets will show up as bands of light.

of detecting strain in glassware. The glassblower should spend some time examining glass known to be strain free and comparing its appearance in the strainviewer with that of strained glass. He will then learn to distinguish between the light and colour effects resulting from normal refraction and those resulting from double refraction (Fig. 5.7).

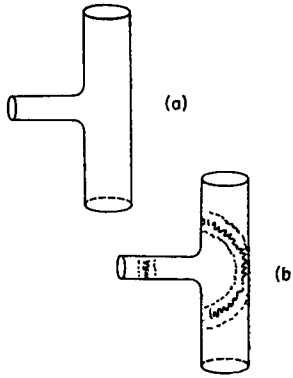

FIG. 5.7. (a) Shows the appearance of annealed and therefore strain-free glass. (b) Shows the light bands in strained glass and indicates the need for annealing.

More elaborate and sensitive commercial instruments are available and one should be installed in all workshops that make glassware, particularly when the apparatus is to be used for hazardous research projects, and the possibility of glass failure must be reduced to the minimum.

Some of these instruments are equipped with a tint-plate, which shows strained regions in a contrasting colour to that of unstrained glass. With a little practice and experience it is possible to tell whether residual strains are likely to cause breakage. In general, bright narrow bands of irregular shape will cause failure and the glassware should be re-annealed.

CHAPTER 6

Elementary Glassworking

RICHARD THRELFAL,[1] in his book *On Laboratory Arts*, said: "The art of glassblowing has the conspicuous advantage from the point of view of literary presentation of being to a great extent incommunicable."

A highly skilled and artistically accomplished glassblower[2] remarked after reading a book on neon sign tube bending: "Only those who can already make neon sign glassware will understand books on the subject."

None the less, an attempt will be made in the following chapter to describe some of the basic operations necessary for making simple laboratory glassware. The reader who combines determination and industry with ingenuity and imagination will make steady progress. An ability to picture the finished article in three dimensions, rather than in two, is of considerable help. He who waits till every step is clearly defined, till every path is obviously signposted, will stand still. Glassblowing is not only a trade, a craft and a profession, it is an art, and mastery of an art needs courage and an adventurous spirit. Art also demands from those who take it up a measure of humility and dedication.

Cutting Glass Sheet

There is a measure of skill involved in cutting up glass sheet that can be acquired with very little practice, provided some care is taken. A good diamond glasscutter is best. This is a personal tool and should not be lent out to all and sundry, as it will almost certainly be damaged. Other types of glasscutter will do if they are in good condition.

Whenever possible, new glass should be used. Old glass from windows or fume cupboards will be weathered and have invisible surface defects that upset attempts at cutting. Such old glass is always difficult, and often impossible, to cut straight.

The required shape should first be drawn in outline, on paper, using set squares to ensure that all angles and dimensions are accurate.

A flat table top is necessary. A few sheets of paper under the paper pattern will help to prevent unexpected breakage if too much pressure is used on the tool.

The cleaned glass sheet is arranged on top of the drawing so that the best edge is exactly in line with the required shape. A straight edge, about 6 mm (0·25 in.) thick, is laid on the glass and adjusted so that the cutting point is exactly over the pencil line (see Fig. 6.1). With one clean, smooth movement, a scratch (or

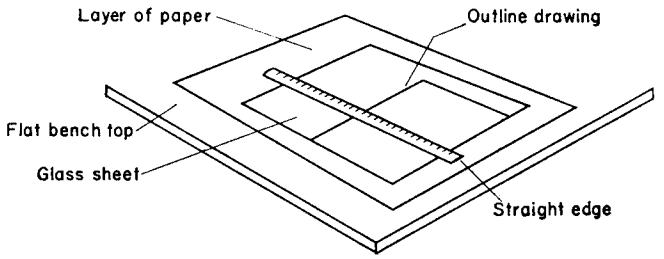

FIG. 6.1. Cutting glass sheet to an outline drawing.

incision) is made on the glass from edge to edge. The sheet is now held as shown in Fig. 6.2 and quickly bent; the glass should break off along the mark.

Some preliminary practice with odd pieces of glass will help to fix the best angle at which to hold the tool and the correct pressure needed to make a good scratch. The glass must be clean. A better result can be obtained if the diamond tool is first dipped in turpentine.

When all the sides have been cut the glass should fit the outline sketch. The edges and corners will be considerably streng-

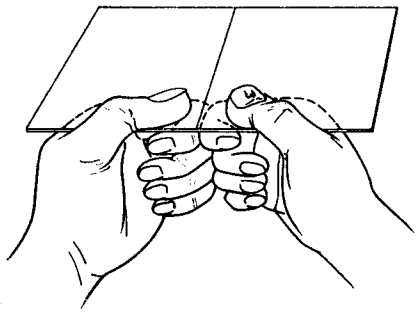

FIG. 6.2. Hand position for a breaking a glass sheet at a mark. The thumbs exert a downward pressure, the fingers press upwards.

thened if they are lightly rubbed with a No. 120 carborundum stone. The stone is stroked along the edges and across the corners, as shown in Fig. 6.3.

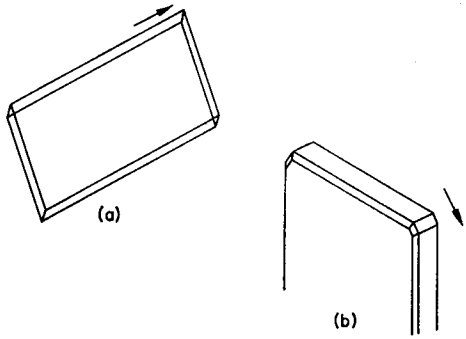

FIG. 6.3. Removing sharp edges with a hand-stone. (a) Shows the edges of a newly cut piece of glass sheet. (b) Shows the edges ground off. The arrows indicate the direction of movement of the stone.

Plate glass may prove difficult to break. It should be reversed on the table and tapped with the back of the tool, opposite the scratch. A small crack will start and can be led, with continued tapping, to the edge of the sheet (see Fig. 6.4).

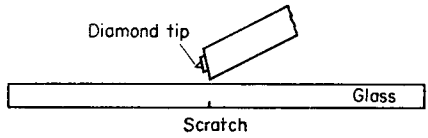

FIG. 6.4. Starting a crack in plate glass. It is essential to make a good deep mark with a sharp tool. The glass is inverted and tapped with the metal edge of the tool directly opposite the mark.

Providing there are no re-entrant curves or angles, any shape, including circles, can be cut out of sheet glass. Tangential scratches are made round curves, one at a time and the waste glass broken off, circles and arcs of circles are best drawn with a diamond compass, which is illustrated in Fig. 6.5.

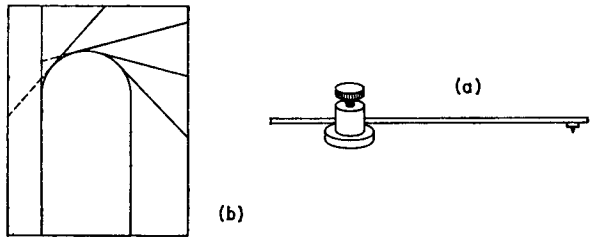

FIG. 6.5. A diamond-tipped compass for cutting curves. (a) Shows the compass with rubber centre and adjustable radius rod. (b) Indicates the tangential marks used to remove the waste glass.

Hints

Practise on odd pieces of sheet. Keep the bench top free from glass splinters which will scratch the surfaces.

Dip the diamond tool in turpentine before each cut. Scratch and break one line at a time. Make only one scratch mark. Find out the correct angle and pressure for the tool. Use new glass. Chip off small pieces of waste with old pliers or side-cutters.

Drilling Glass Sheet

Holes up to 12·5 mm (0·5 in.) diameter can readily be drilled in glass sheet with tungsten carbide tipped tools. The tool manufacturer's instructions should be followed carefully. Either a drill press or a wheelbrace can be used; some practice is required to decide the best pressure to use. The drill operates in a pool of turpentine kept in place with a plasticine or rubber ring. When the point of the tool is halfway through, or a little more, the glass is inverted and drilling continued from the other side (see Fig. 6.6). These drills can be sharpened on a suitable grindstone. The angles of the cutting edges are fairly critical and should not be appreciably altered.

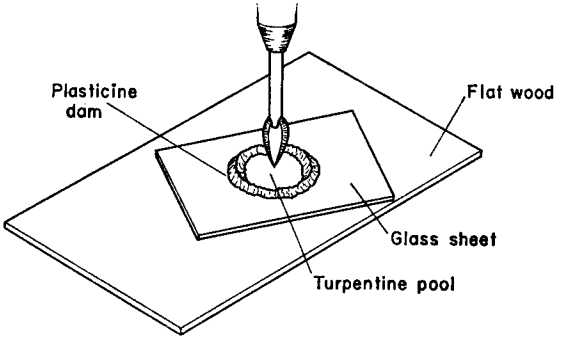

FIG. 6.6. Cutting a hole in a sheet glass.

Brass tubes of suitable diameter, turning in a suspension of 220-mesh carborundum powder in water containing about 10% glycerine, will also cut holes in glass sheet. This is best done on a drill press with a piece of flat wood on the press table. The drill should be raised every few seconds to allow fresh carborundum to flow under the tube edge. Again the glass must be inverted before the drill cuts through the bottom surface.

This second method is somewhat more reliable than the first, but much slower. Holes of similar size can also be drilled in bottles and jars. The bottle must be supported on wooden V-

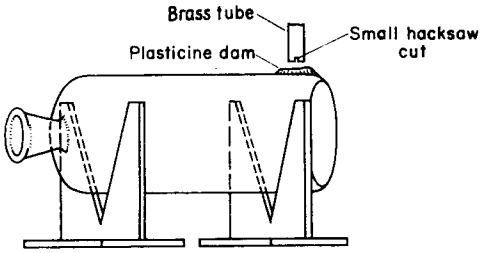

FIG. 6.7. Cutting a hole in a bottle. The small cut in the brass tube helps to retain carborundum grit.

blocks and very gentle pressure should be applied to the tool when it is about to penetrate the bottom surface. (See Fig. 6.7.)

Basic Operations in Soda Glass

Cutting glass tube and rod. Glass tube up to about 15 mm diameter and rod up to 8 mm diameter can be cut with a glass knife as follows: a single clean mark is made on the outside surface of the tube, extending about one-third of the distance round the circumference and at right angles to the centre line (Fig. 6.8). The tube is now held firmly in both hands, with the knife mark midway between them; an even pressure is applied with the hands, combining pulling and slight bending. The tube should separate cleanly at the mark (see Fig. 6.9). This operation is made a little easier if the mark is wetted with a finger tip. Saliva will serve. The glass-cutting knife must be sharp. If any difficulty is found in making the mark, examine the knife edge. Resharpen on a stone as described in Chapter 4 if it should prove blunt.

If a file is used it must be in new condition or should have had the cutting edge recently renewed as described in Chapter 4. The file will cut a notch in the tube with one single stroke. The tube should be separated without delay. Some heat is generated by the file as it moves rapidly across the glass. This heat helps to start a small crack. Sometimes, in spite of all precautions, a

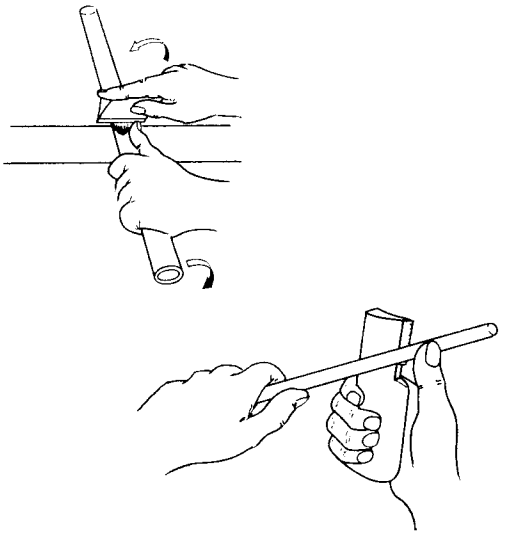

FIG. 6.8. Alternative methods of making a knife mark on glass tubing and rod.

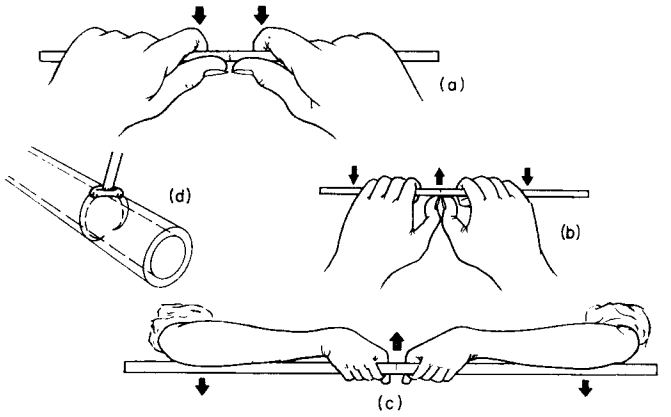

FIG. 6.9. Hand positions for breaking tubing and rod. (a) A method of breaking small diameter glass tubing. (b) A method of breaking rod. (c) Hand and forearm action for breaking large tubes. (d) Applying hot glass to knife mark.

length of tube will not cut straight. Examine the tube end for uneven wall and make a knife mark on the thickest part. Should the glass still break unevenly instead of cutting straight, then the glass tube has been left strained and should either be annealed or discarded. Such strained tubes are rare and no tube should be discarded unless its behaviour is very different from that of others of the same size. The first few used are unlikely to be so strained.

A sawing action with either knife or file is unnecessary, and will only blunt the tool; one single straight mark made with a smooth movement of the hands is all that is required. The knife or file must be sharp. If considerable pressure is required to mark the glass, the tool is blunt and must be resharpened.

The ends of all glass tubes, as taken from the stock, should be examined. Unless they are perfectly straight, free from cracks (no matter how small) and perfectly clean, they must be cut off and discarded. Make a single knife or file mark round one-third of the circumference and 3 cm from the end. Now take a piece of soda glass rod, about 3–4 mm diameter and some 15 cm long, and heat one end in a small hot flame until the glass just melts. Hold the tube in the left hand, the rod in the right hand and apply the hot blob of glass to the knife mark. If the mark has been made properly, with a sharp knife, then a crack will appear in the glass and the end can then be removed. Such a crack can be led round glass tubes of quite large diameter if the rod is repeatedly reheated and applied to the knife mark, just ahead of the crack.

Laboratory glassblowing consists essentially of heating a length of tube until it is plastic, then changing its shape by pulling, pushing, or bending, by blowing with the mouth, or by the application of a suitable tool. These changes of shape must be made with the speed, certainty and confidence that come from experience, and must be completed in the very short time that elapses before the molten glass cools to the temperature at which it sets.

A number of basic techniques must be practised, and a fair degree of perfection in their application achieved, before simple glass apparatus can be made. Such apparatus must be not only functional, but also reasonably strong and reliable. It will also

be more pleasing to use if it has a neat, well finished appearance. To achieve a reasonable degree of perfection it is necessary to repeat over and over again the exercises that follow. When an acceptable standard has been reached the exercises should still be performed, either as exercises or as part of the work involved in making glass equipment.

This continued repetition must be undertaken with close attention to detail, and with single-minded determination to improve the standard of the work and reduce the time spent on any operation. There may be some temptation to make fairly elaborate glassware before the basic techniques have been mastered. Such premature attempts are doomed to failure, since it is impossible to make, say, an all-glass condenser before skill is acquired in drawing points, in preparing and attaching water inlet and outlet tubes, in constricting, flanging, making internal seals and in the important technique, annealing.

Indentured apprentice glassblowers normally spend 5 years learning this work. Allowing 50 working weeks in a year and 40 hr in a week, we have apprentice glassblowers spending 10,000 hr at the bench. In general, reasonable competence is reached in one year, or 2000 hr; the remainder of the time is necessary for broadening experience and increasing efficiency. Clearly this order of time is not available to laboratory technicians, students or other laboratory workers who have other skills to acquire and a wide range of theoretical knowledge to absorb. These figures are not set down to frighten off would-be glassblowers, but rather to impress them with the need for extra effort and concentration in reaching an acceptable standard of work in a comparatively short time. It follows that technicians and others who take up laboratory glassblowing, either as a useful and satisfying skill or as a required subject in an approved course, must have access to glassblowing facilities and must use every possible opportunity to practise the basic techniques. Glassblowing cannot be learned simply by reading a description of the techniques in a book; the reading must be supplemented by practice and, wherever possible, by the personal supervision and guidance of a skilled craftsman.

FINISHING OPEN TUBE ENDS

No piece of glassware, however simple, has been properly finished if any of the tube ends are left with sharp edges. Such edges are fragile, particularly those of large diameter, and they will cut into and damage cork and rubber stoppers, rubber tubes and, not infrequently, fingers, hands and lips. This important operation will be used as an introduction to the use of the left hand.

Prepare a number of glass tubes 30 cm long and from 10 to 12 mm diameter. Carefully measure each one before marking with the knife and cutting from the length. Should the glass break unevenly or show any sign of cracks, then cut a new straight end before proceeding. It is most unwise to mark off the whole tube into 30 cm lengths before proceeding to break off each one. The action of breaking off one short length can cause others to break at the marks. They will then fall to the floor and be damaged. A cork stopper is selected to fit the tube in use, the stoppered tube is held in the left hand, palm down (see Fig. 6.10).

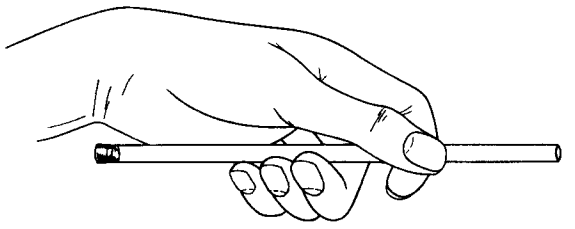

FIG. 6.10. Tubing ready for rotation in the left hand. Note the positions of the fingers and thumb.

This stopper has several functions. It prevents the flame from entering the tube, passing through it and heating it. If the flame cannot pass through the tube then the amount of water vapour condensed on the inside wall of the glass is considerably reduced. This water vapour is a product of the combustion of the fuel gas. If it is allowed to accumulate it may run down on to the hot

glass and crack finished or almost finished work. When blowing is involved, in later exercises, it is essential that only one tube have an open end. All others, if there are more than one, must be stoppered. The habit of stopping up these ends should be acquired as soon as possible.

Rotate the tube by rolling it on the pads of the index finger and thumb. The other three fingers should be curved round the tube, holding it very lightly. The angle through which the tube rotates with each rolling movement will depend on the flexibility of the fingers and also on the diameter of the tube. For 10 mm tubes it should be 200° or more than half a turn. This apparently simple operation requires much practice, as it is an essential basis of all bench glassblowing. The tube should be so held that its mid-point is somewhere in the left hand; it should be rotated clockwise when viewed at the open end. After each rolling movement of the thumb and forefinger they are removed from the glass and a new rolling movement started. The rotation, although intermittent, should be as smooth as possible. The open end should not wobble about, or it will not remain in the flame when heated. The result will be uneven melting and a poor job. The tube end is now brought into a soft gas flame with very little air flowing. Rotation should continue steadily and the glass must be kept in the flame until it shows a sodium yellow colour on the edges. The air and gas are now adjusted to give a small hot flame, about the same diameter as the tube, i.e. about 12 mm. As soon as the tube end begins to melt it is removed from the flame and set upright in the cooling rack. On no account must the hot end be laid on the bench top or allowed to cool in contact with anything but air. This procedure is repeated for one end of each tube in turn, then the other ends are similarly fused and set up to cool.

In each case uniform heating must be continued till the edges are seen to just melt. If the heating is insufficient, the edges will remain sharp and nothing will be achieved. Should the heating be prolonged, the edges will melt inwards and the tube will be constricted unnecessarily.

This procedure should be repeated over and over again, to the point of boredom. The left hand and fingers will be gradually

strengthened and made more flexible. Close attention to steady rotation and to keeping the tube end in the hottest part of the flame must never be relaxed.

It cannot be over-emphasized that glassblowing is a highly skilled craft, and that the foundations of future skills are laid when simple techniques are learned. Mastery of such techniques will come only to those who bring all their faculties to bear on the work in hand.

The manipulation of glass in the flame brings muscles into play that are, at first, unaccustomed to prolonged and precise movements. Practice must be continued until this manipulation becomes automatic. A number of exercises will be described in which the glass is held in and rotated by the left hand. On no account should the right hand be used. The main objective is to train and exercise the left hand in steady rotation and control of glass tube. All glassblowing requires co-ordination of both hands; the left hand does, so to speak, the donkey work. It turns the main part of the job and keeps the worked glass in the flame. The right hand is then left free to control wall-thickness, to do the pulling or pushing, to make bends and bulbs, to handle tools, and to adjust the burner gas- and air-taps.

All those who take up glassblowing for the first time will be faced with many difficulties. Some will be bored with the constant repetition so necessary at the beginning. Some will be frustrated by their inability to control their hand and finger movements. Some will be infuriated by the apparently unforeseeable and unco-operative behaviour of molten glass. Do not be discouraged. Instead, examine a hollow key vacuum stopcock, or a tilting McLeod gauge. The men who made such glassware had the same difficulties now being experienced by you, and overcame them by continued practice, by the pursuit of perfection, by determination not to be beaten, and, finally, by taking pleasure and a deep satisfaction from making something both beautiful and useful. There is beauty in a straight-cut and properly fused tube end, in a hemispherical test-tube end of uniform wall-thickness, in a smoothly curved bend and in a perfectly straight, almost invisible join.

Open tube ends can be further strengthened if the heating is prolonged so that the glass becomes quite molten, and because of its surface tension gathers back into a thickened bead (see Fig. 6.11a). At this stage the tube is removed from the flame

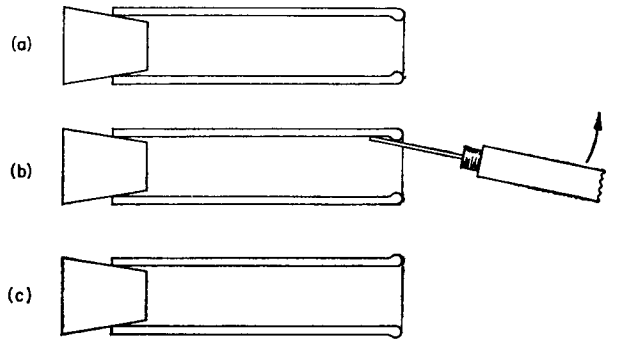

FIG. 6.11. Making a rim on a tube. (a) The heated glass thickened and constricted. (b) The tube end being opened out with a reamer. (c) The finished reinforced end ready for flame-annealing.

and the small pointed brass tool, already heated slightly, is inserted inside the tube till the point lightly touches the cool, unmelted glass wall. The tube is rotated back and forth, through as large an angle as possible, and meanwhile the tool is also turned in the opposite direction. The molten glass rim should be quickly but gradually spun out till the inside diameter is again the same as that of the tube. The worked end and surrounding glass are now annealed in a soft flame. All glass worked with tools will be quickly and locally cooled and must therefore be annealed in the flame. The tube and brass tool can both be controlled more easily if the tool point touches the inside cool wall. The rim should be opened out gradually, that is, not in one movement. The whole tooling operation takes very little time, as the molten glass is quickly cooled and soon becomes unworkable. At the instant when the glass is changing its rotation from clockwise to anti-clockwise it will be stationary. At this instant it is important that the tool be turned slightly, otherwise a small dent will

be made in the soft glass. Again, practice is necessary and at least 100 tube ends should be reamed out, as described, after reasonable proficiency has been acquired. Progress will be accelerated if close attention is paid to the hand positions and movements of a skilled glassblower. All elementary techniques are more easily learned by copying those of experienced men. Good hand positions and movements must be deliberately used from the beginning, as they are more difficult to learn if faulty or unsuitable methods have first to be unlearned.

Tube ends can be made to fit cork or rubber stoppers by reaming them out to approximately the same taper as the stopper. When the reinforced rim has been made, as described above, the tube is put back into the flame with slightly reduced air-flow. Heating is continued until about 2 cm of the tube length is plastic. The glass is taken out of the flame and the same heated tool inserted so that the point touches the inside wall (Fig. 6.12a).

The tube and tool are turned as before till the end is slightly opened out. There is no time to lose as the glass quickly sets hard.

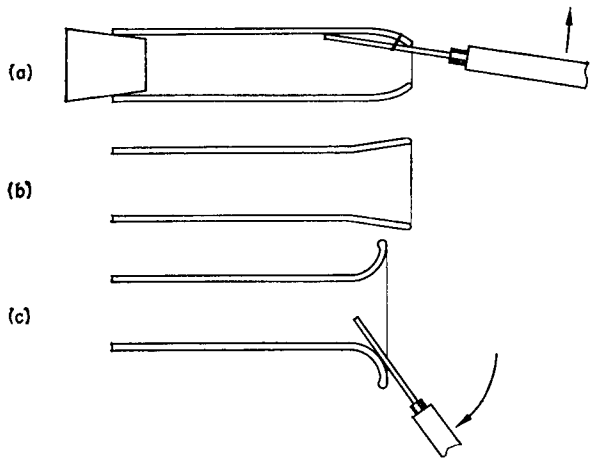

FIG. 6.12. Flaring and flanging tube ends. (a) The tool is moved away from the tube centre-line to flare the plastic glass. (b) Finished flared end. (c) A flanged end, made by taking the methods of (a) a step farther.

The tube should now be shaped as in Fig. 6.12b. Uniform heating is essential for this operation and much practice will be required. Soda glass must always be pre-heated in a flame with very little air flowing, or the tube may crack and be spoiled. The air-flow can then be increased and the flame adjusted to melt the glass.

Tubes are frequently required with flanged ends, for example the inner tubes of all-glass condensers are flanged out before sealing to the water jacket. Flanges are made exactly as above, except that the glass is spun out with the tool to the shape shown in Fig. 6.12c. Flanges of this type are best when bell-shaped, i.e. curved in section. The end of the flange must be flat and circular, in a plane at right angles to the centre line of the tube.

It is worth noting at this stage that the last two operations, i.e. tapering and flanging, should be carried out promptly, and with, wherever possible, no reheating of the glass. Such reheating will result in too much glass being melted, while repeated efforts to improve an irregular taper or flange will lead, in most cases, to greater irregularity.

Much practice is, of course, required before these techniques are mastered.

Pulling Points

These very convenient aids to glassblowing are known as points, or spindles, or splints and sometimes as spears (Fig. 6.13a). Both hands are used, and good co-ordination of their movements must be developed by continued and determined practice.

Use the 30 cm lengths of tube, the ends of which are already fused. Make a small mark at the centre of each tube with a glass-marking pencil, or, if preferred, with a glass knife or file. Now make two other similar marks on the tubes, one on each side of the first mark and at a distance from it equal to the diameter of the tube. The three marks should be in line (see Fig. 6.13b).

The first tube is held with the left palm down as in the previous exercises. The right palm faces up and the tube is held between

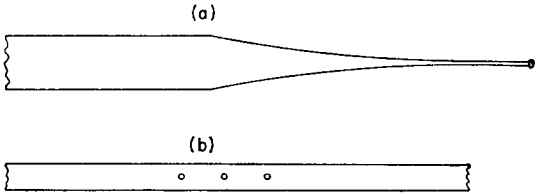

FIG. 6.13. (a) A point on a glass tube. (b) Shows guide marks which help to keep the tube free from twists.

the finger and thumb, the other fingers being arranged so that at least one is under the tube and ready to support it when the finger and thumb release their hold and take a new hold of the tube (see Fig. 6.14).

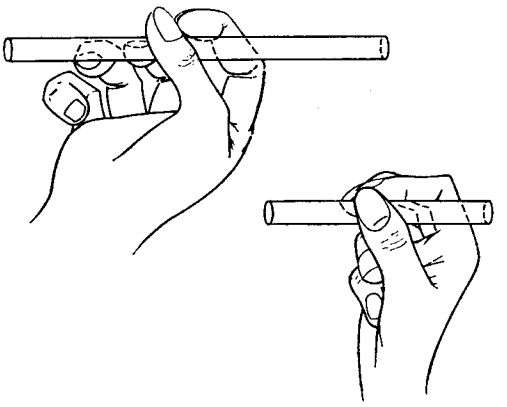

FIG. 6.14. The right hand. Note alternative finger-and-thumb positions.

After some preliminary practice in rotating the tube with both hands (so that there is no tendency to bend the glass, to stretch it or to push it together), a first attempt at pulling points is made. The glass is heated in a soft flame with just enough air to completely remove all yellow colour. The centre mark must be in the middle of the flame. As soon as the glass is plastic a gentle pressure is applied by the right hand, towards the left hand, so

that the plastic glass is pushed up into a very slight bulge. Heating is continued (see Fig. 6.15) with steady rotation of the whole tube. Pay close attention to the three marks, keeping them always in line. The marks serve as a guide to prevent the glass being twisted. Keep the whole tube straight and avoid any tendency to stretch the plastic glass or to push it together too much.

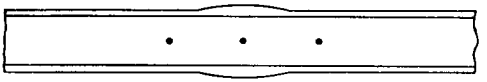

FIG. 6.15. Pulling points. The glass is shown hot and gathered ready to pull out.

The left hand maintains steady and uniform rotation. The right hand keeps the tube straight, and free from twists.

When all the glass between the two outside marks is seen to be quite plastic, remove the tube from the flame and slowly draw it out into a long constriction. The tube must still be all in line. The shoulders, where the original tube begins to taper, should be from 30 to 35 cm apart just as the glass begins to set.

Complete rotation will be difficult to combine with steady pulling while the tube is kept straight, so a back-and-forth rotation can be substituted. In any event the tube must be turned to prevent the hot plastic glass from sagging towards the bottom and to help to keep the points straight. Rotation and pulling must be continued until the glass has completely set. Many beginners succeed in making straight points, then allow the still plastic glass to get out of line almost at the very last moment.

The drawn out part of the glass may be cut in the centre with the knife. Usually the glass is melted in the centre and pulled apart, the ends being then fused into small round blobs (see Fig. 6.16).

There is some temptation to hold both hands with the palm down—indeed, this appears at first sight to be the logical way to hold the glass. However, when blowing is involved (as in joining tubes and blowing bulbs in later exercises) it will be found more convenient to bring the open end (held in the right hand) to the

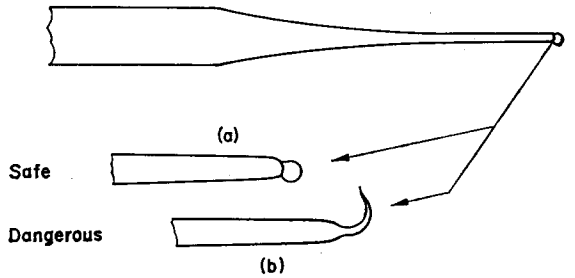

FIG. 6.16. Safe and dangerous points. The end of the point should be melted into a round blob as shown in (a). Ends left as shown in (b) can be very sharp.

mouth if the right hand is held palm up. It is therefore necessary to learn to control and manipulate the hot glass with the right hand held as described above, rather than with the palm down.

When all the 30 cm by 10–12 mm glass tubes have been drawn out, and are cool, they can be set aside ready for another exercise.

Point pulling is not easy and many hours must be devoted to practice exercises. Further, the hands must become accustomed to pulling points under conditions different from those set out above. With this need for versatility in mind the following exercise is described.

Balance a 150 cm length of 12 mm o.d. tube on the right forefinger to find the mid-point, then cut the length in half. Cut three or four 15 cm pieces of 5 mm rod and place them to the right of the burner, and within easy reach (see Fig. 6.17).

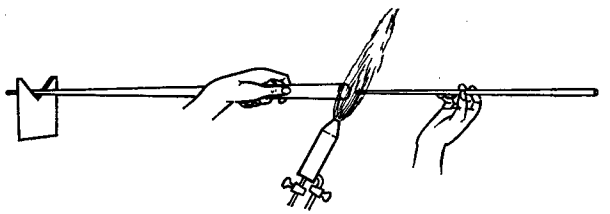

FIG. 6.17. Pulling a point on a tube end. Showing the tube rest, hand positions, burner flame and rod handle.

With the half-length in the tube support, melt the end until the glass becomes plastic and just begins to close in. At the same time heat one end of a piece of the rod until the edges just begin the fuse. The rod must, of course, be rolled between the pads of the thumb and forefinger and kept steady in the flame. Care must be taken to avoid premature contact between the rod and tube ends. The tube end should be much hotter than the rod; it should be plastic while the rod need only be fused.

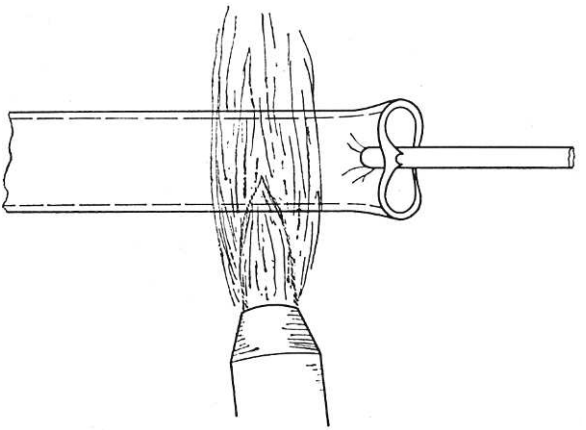

FIG. 6.18. The attachment of the rod handle to the plastic tube end.

The rod is now quickly pressed against the inside of the tube, where it will stick, and immediately pressed over to touch a point diametrically opposite. The glass is removed from the flame. Rotation is momentarily stopped for this manipulation of the rod, but must be restarted at once. The rod is now centred with respect to the tube, by rotating both and by gentle pulling (see Fig. 6.18). This centring of the rod is important as it will be used for pulling a point. If the rod is eccentric, then the point will also be eccentric and hence unsatisfactory. Heating is now transferred to a region about 2·5 cm from the tube end, and a point pulled out exactly as before.

This procedure is repeated, using successive completed points to pull out the glass, and leaving 2·5 cm of undisturbed tube each time, until the whole length has been used up (see Fig. 6.19). All double points which are straight, strong and of usable length should be set aside for a later exercise.

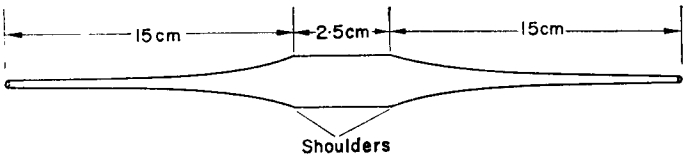

FIG. 6.19. Double points, showing the shoulders. Both points should be concentric with the tube.

It is strongly recommended that this exercise be continued, using tubing of progressively increased diameter, until serviceable double points with a predetermined length between the shoulders can be drawn out in tubing up to 25 mm o.d. Remember to melt a length of tube equal to twice the diameter, and to continue turning and pulling till the glass has set.

Test-tube Ends

In making test-tube ends always begin by pulling good straight serviceable points. The points in this case will be discarded immediately, none the less an opportunity to pull good points should never be missed. Further, poor points leave an irregular shoulder which will add to the difficulty of blowing a nice round hemisphere.

Use the 10–12 mm diameter tubes with fused ends and already drawn out. After a preliminary heat up in a soft flame, readjust the gas and air-flow to obtain a small pointed flame. Be careful not to use too much air; the blue inner cone should be clearly seen. Heat the point close to the shoulder, with continuous rotation, till the glass melts and constricts as in Fig. 6.20a. Remove the glass from the flame and pull out to a very thin, long, point

(see Fig. 6.20b). Melt this point off as close to the tube shoulder as possible as in Fig. 6.20c. There will now be a small bead of glass left as shown at *x* in Fig. 6.20c. If the bead is too big then some more glass must be pulled off. Skill in pulling off just the right amount of glass can only be acquired by experience. In general it can be assumed that the bead should be as small as possible without actually making a hole in the glass. The end of the tube is now melted down with a slightly larger and softer flame, till it is shaped as in Fig. 6.20e, and blown to a hemispherical shape as in Fig. 6.20f. Heating should not be allowed to extend beyond the shoulder, otherwise too much glass will be melted and the end, when blown out, will be shaped like Fig. 6.20g. The wall-thickness of the test-tube end should be the same as that of the tube. If left too thin it will be mechanically weak. If too thick, it may be thermally weak and liable to crack when reheated at any subsequent time. This wall-thickness, and all others, must be closely observed when the glass is plastic, and being blown or otherwise worked. The ability to observe and control wall-thickness should be developed as soon as possible.

Blowing

Blowing technique has already been described in Chapter 3 since there is a minor hazard involved. In addition to the need to have the glass hot enough to be quite plastic it is also necessary to have it under close observation. Wall-thickness, tube or bulb diameter and reasonably accurate alignment cannot be recovered if the glass being blown is out of sight.

Flat Ends

Proceed again to the stage indicated by Fig. 6.20d and remove rather more glass without disturbing the shape of the shoulder. Reheat the small bulge in a moderately hot flame and gradually melt all the glass back to the shoulder. The tube end should now

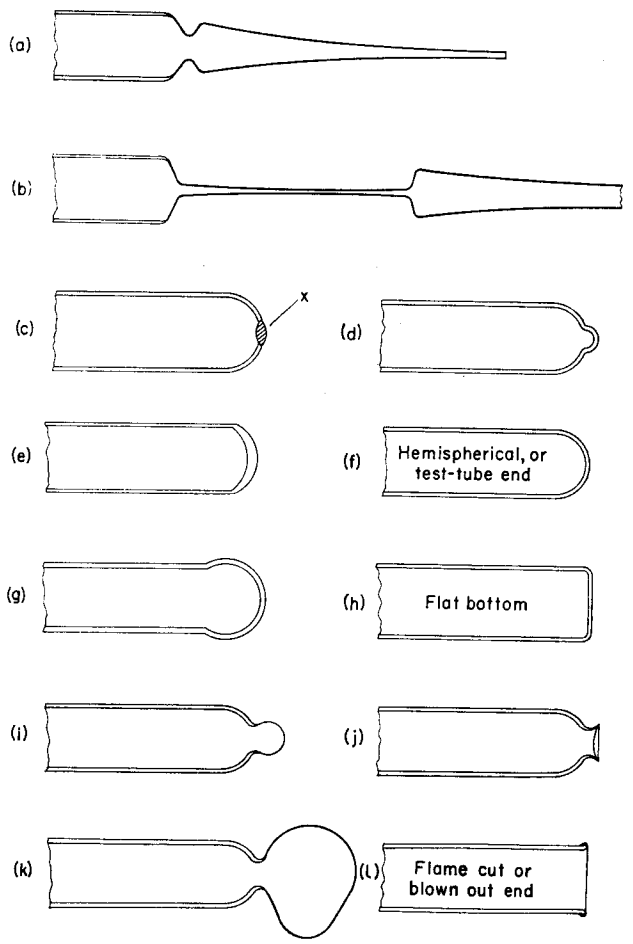

FIG. 6.20. The steps required to make test-tube ends, flat-bottomed ends and blown-out ends.

appear as shown in Fig. 6.20h. A number of attempts will be required to decide just how much glass to leave beyond the shoulder, so that the flat bottom has the same wall-thickness as the tube. Some difficulty will be experienced in making flat ends if the wall of the tube is uneven, or if the end is allowed to wobble about, in and out of the flame. This difficulty can be overcome to a certain extent by gently blowing out the molten glass and immediately pressing the tube end against a flat carbon plate or brass tool. The tube end should then be flame-annealed and set up to cool.

Blown Out or Flame Cut Ends

Proceed as in the previous exercise to the stage indicated by Fig. 6.20d. With the same flame reheat the small bulb and blow it to the shape shown in Fig. 6.20i. Allow the glass to cool slightly, then reheat at the end of the bulb, flattening it to the shape (Fig. 6.20j) by melting.

When the lens-shaped blob of glass is turning true with the centre line of the tube, blow it out quickly to a very thin bulb (Fig. 6.20k). Remove this bulb, which will be very thin and fragile, and scrape the edge of the open end until it is reasonably straight, then remelt and open it out with the small tool till it has the same bore as the tube, as in Fig. 6.20e.

Bends

Glass rod up to about 8 mm diameter can be readily bent into a variety of shapes. The flame used should be of sufficient size to melt enough glass for the required bend. It should be a soft flame with very little air.

All bends can be kept under control, and so will have a good shape, if the plastic glass is bent upwards and kept in a vertical plane (Fig. 6.21). The limbs of the bend must be kept parallel and in the same plane by repeated inspection and adjustment as

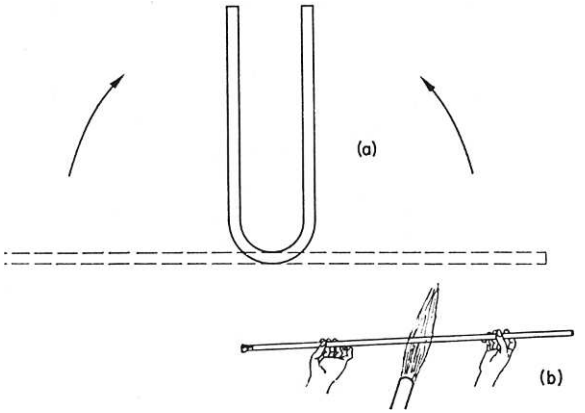

FIG. 6.21. Bending rod and tubing. (a) The heated rod is bent upwards in a vertical plane. (b) Hand position for heating rod and tubing as a preliminary to bending.

the glass cools and sets. The rod need be heated till it is just plastic and no more. It must not be stretched or deformed in any way. A very gentle pull, just before bending starts, will remove any misalignment of the rod. U-shaped bends are somewhat easier to make if both hands are held with the palms facing up. This avoids changing the hand-hold when the rod limbs reach the vertical position. Rotation is done with the forefinger and thumb as before; the rod is supported by the palms when the hold is renewed. Bends of large radius, that is 20 mm or more, can be done in one heating if the rod is held along the flame as shown in Fig. 6.22. Care must, of course, be exercised to keep

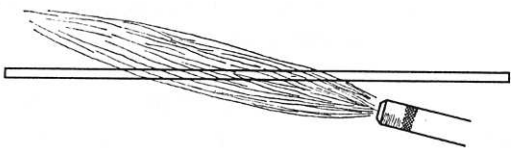

FIG. 6.22. To heat a suitable length of tubing or rod for a large radius bend, the glass is held and rotated in the flame as shown.

the left hand below the flame and a longer than usual rod must be used. It is often better to make such bends standing up, with the burner turned round so that the flame is almost parallel with the front edge of the bench. Again, very little air is required and the rod should be straightened with a gentle pull before being bent up in a vertical plane. Some adjustments to the longer radius bends may be required. Local reheating should be done with the same flame and frequent inspection should accompany the adjustments. The natural tendency for long bends to fall into a shape other than an arc of a circle can be corrected by moving the vertical limbs slightly apart. Such movement should, of course, be accompanied by frequent continued inspection. (See Fig. 6.23a.)

All bends must be made upwards in a vertical plane, otherwise the plastic glass will not remain in the plane of the limbs but take up a shape such as Fig. 6.23b.

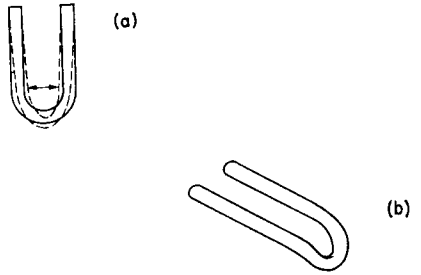

FIG. 6.23. Some faults to be avoided. (a) Large radius U-bends tend to sag downwards in the middle. The shape may be improved by gently moving the limbs away from one another, as shown by arrows. (b) The probable result of bending both tube and rod in a horizontal plane.

It will now be apparent that good judgement of shapes and angles must be possessed, or acquired, and applied without delay. Good serviceable bends can be made in tubes of small diameter by exactly the same methods as those described for rod, provided always that the radius of the bend is not less than 5 times the diameter of the tube.

Similarly, capillary tube can be bent to quite a small radius. Preheating of the glass must proceed rather more slowly. It should start with no air-flow into the gas flame. When the bend is finished, annealing should be very thorough: the air-flow at the beginning must be quite small, and should be reduced to zero when the sodium yellow colour appears on the surfaces surrounding the bend.

During this necessarily prolonged annealing the bend and limbs should be inspected at frequent intervals and the shape adjusted as required.

Sharp Bends

Sharp bends of small radius and with 90° or less between the limbs must be blown to recover the circular cross-section of the tube. The procedure for tube up to about 15 mm o.d. is as follows: the selected tube is stoppered at one end and marked in the centre with the glass knife. Such marks can be quite small, they will not disappear when the glass is heated, but will melt in. The finished bend will not be weakened in any way by such treatment. Pencil lines, on the other hand, have a tendency to burn off and disappear if they are small, or leave large disfiguring marks if they are big.

Heat a length of tube equal to twice the tube diameter in a medium flame until the glass wall is slightly thickened and constricted. Remove the glass from the flame, pull it very slightly and then bend it up to the required angle. Blow into the open end with gradually increasing pressure till the tube recovers its original diameter. (See Fig. 6.24.) The degree of constriction and thickening of the tube wall is quite small. The melted glass must be really pliable and quite free from twists. It should be uniformly hot and have no excessively thin or thick regions. Practice exercises should be continued and repeated from time to time till smooth bends of good shape and uniform wall-thickness can be made. Some faults in bends are shown in Fig. 6.25.

The glass on the outside of the bend will be stretched to a greater length than that on the inside and will tend to be thinner

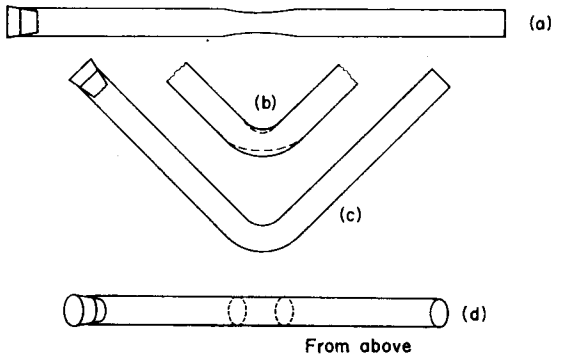

FIG. 6.24. Short radius bend. (a) The glass heated and constricted slightly and ready to bend; (b) the dotted lines show the tube after bending and before blowing up; (c) and (d) show the finished bend in plan and elevation. Note that both limbs and the bend lie in the same plane.

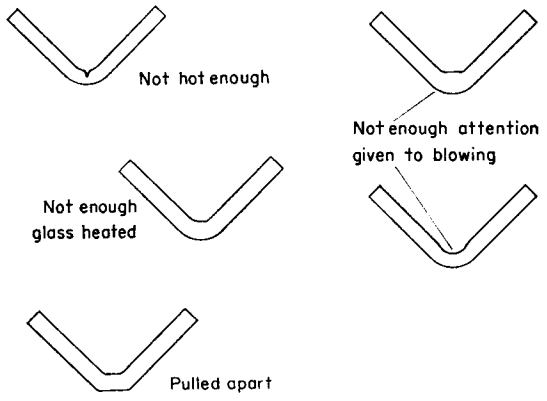

FIG. 6.25. Some other faults in short-radius bends.

in the wall. This can be partly offset by slowly and steadily increasing the air pressure when blowing. Poor bends will follow insufficient heating of the glass, or heating too short a length of tube, or failing to allow the glass wall to thicken slightly or by failing to heat the required glass uniformly (see Fig. 6.26).

Joining Rods

The ends of the rod should be quite clean and preferably cut straight. After a preliminary heat up in a soft flame the ends are brought close together and heated in a small pointed flame, about the same diameter as the rod, or a bit smaller. The air-flow is adjusted to give a distinct inner blue cone, as in Fig. 6.26a.

When the ends are melting and just beginning to gather into molten blobs, they are pressed gently together and immediately pulled very slightly apart. The join should now appear as in Fig. 6.26b. Heating is continued till the molten glass gathers up

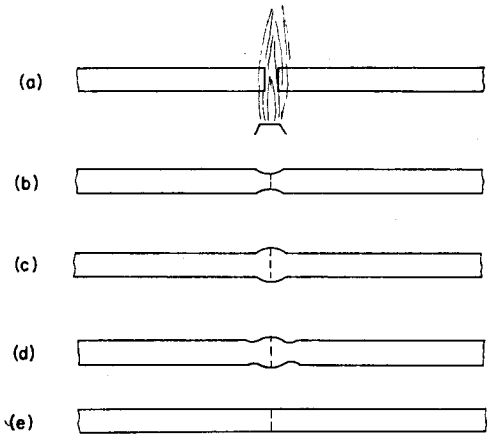

FIG. 6.26. Making a join in rod. (a), (b) and (e) show the successive steps when the minimum glass is melted; (c) and (d) show the result of melting more glass than is necessary and of pushing this glass into a large blob.

and is about 1 mm greater in diameter than that of the rod. The glass is now removed from the flame and very slowly pulled out till the diameter is uniform.

This is a very delicate operation and calls for maximum attention to a number of details. The rods must be turned uniformly throughout and they must be kept in line. The molten glass must not be pulled out too far, and therefore made too small in diameter, at any stage. Neither should the molten glass be allowed to gather up much beyond the recommended size, nor should heating be continued after the rod ends are well melted together. If a form such as Fig. 6.26c is made, then on pulling the molten glass out, the result will almost certainly be something like Fig. 6.26d, and much reworking will be required. Rotation with very gentle pulling must be continued till the glass has completely reset. Normal flame-annealing should follow.

Joining Capillary Tube

Capillary tubes of 2 mm bore or more are joined by the same method as that used for rod. Added attention must be given to the bore. When the tube ends are being melted before bringing them together the bore must on no account be allowed to close or constrict appreciably, yet the tube ends must both be sufficiently melted so that the glass flows together when the ends are touched. The ends must be pressed together very gently indeed, so that the bores are exactly in line. The end of the capillary in the left hand is, of course, closed with a rubber plug.

Heating in the small hot flame is continued till the ends are well melted together and the capillary bore has constricted by about 0·5 mm. The open capillary is quickly brought up to the mouth and a steady air pressure applied; the two tubes must be kept in line and turned back and forth while blowing. The molten glass must be kept under close observation and as soon as the bore is very slightly greater than that of the capillary, the glass is slowly pulled out to restore the outside and inside diameters.

Great care must be exercised to avoid melting more glass than

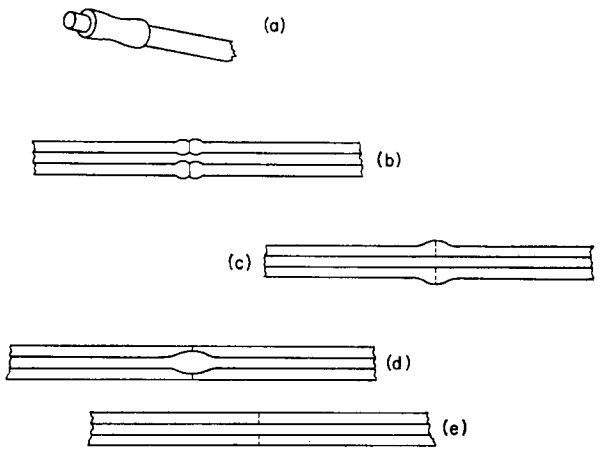

FIG. 6.27. Joining capillary tube of 2 mm bore or more. Note the stoppered rubber tube plug (a). (b) Shows the capillary tubes immediately after they have been brought together and just before they have been very slightly pulled apart. At (c) the glass is ready to blow and pull into a uniform tube (e). (d) Shows the result of excessive blowing.

is absolutely necessary to effect the join. The bore of the melted glass must not be allowed to constrict by more than about 0·5 mm, nor should it be blown up to more than 0·5 mm above that of the capillary. Annealing follows the normal practice. (See Fig. 6.27.)

Joining Tubes

Tubes of all diameters are joined by the same procedure as those just described. It is not, however, advisable to attempt to join tubes much above 18 mm diameter until considerable experience has been gained. Rather more freedom may be taken than with capillary tube when melting the edges together as the bore is unlikely to close.

The tube in the left hand must be stoppered and the end to be placed in the mouth must be free from sharp splinters and pre-

ferably fire-polished. The length of glass melted should be limited to about the diameter of the tube.

Since blowing and pulling both reduce the thickness of the melted glass this must be allowed for. Therefore the glass must be gathered up slightly before the final pulling and blowing are undertaken (Fig. 6.28).

Tubes about 10–12 mm o.d. are perhaps the easiest to handle and a very large number of joins should be made with frequent stops to examine progress and study any faults that may have occurred. The aim should always be towards perfection; it may

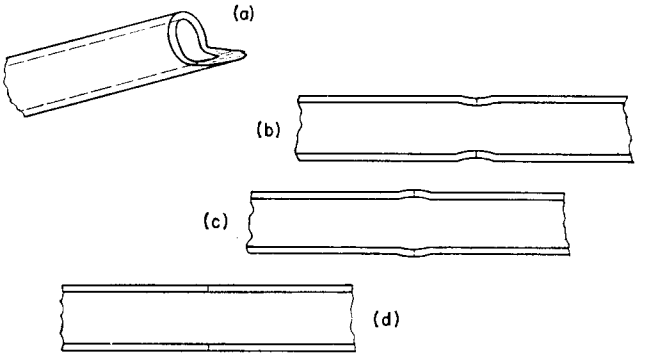

FIG. 6.28. Joining normal tubing: (a) shows the type of sharp edge that may cut the lips; (b) the tube ends pulled very slightly apart after first bringing them together; (c) the join is shown melted thoroughly, slightly gathered and ready to be blown and pulled into shape as shown in (d).

take a long time to achieve, but the hope of attaining it must never be abandoned. Again, watch an experienced glassblower at work, study minutely the positions and movements of both hands, the shape and wall-thickness of the melted glass. The blowing, pulling and straightening techniques should be very closely studied and, as far as is possible, copied.

Spotting

The worked glass must be really hot to make good joins—it will then have the consistency of cold treacle. Such hot glass is difficult to control. It twists, sags, stretches and goes out of line all too easily, and the beginner thinks he will never succeed in making a straight strong join.

A technique known as spotting will be described which will give good results if handled with care.

When the tube ends have been heated to softening point, pressed lightly together and pulled very slightly apart, the burner is re-adjusted to give a small, hot, pointed flame. The join is now heated on one small spot till the edges fuse together completely. This spot is then blown up to the line of the tube wall. Another spot, diametrically opposite the first, is now similarly melted and blown into shape. Spotting continues till the whole circumference of the join has been melted in. If the tubes are above 15 mm o.d. or if the initial join has been very imperfect, it may be advisable to warm up the whole circumference occasionally. Simply turn the gas up a bit and heat all round the join. Thorough annealing accompanied by straightening completes the job. If a small gap, or hole, is left when the tube ends are brought together, do not interrupt heating and rotation. When the worked glass is sufficiently pliable the tubes can be quickly bent with the gap on the inside of the bend. It will then be closed with a little manipulation of the tubes. The glass must be quickly straightened again and heating and rotation resumed.

T-joins

From 10 mm tube cut a 15 cm length and an 8 cm length. Fire-polish both ends of the long piece and only one end of the short one. Stopper one end of the 15 cm length and mark it in the centre. After a preliminary warm up in a soft flame, heat the mark with a hot flame, about half the diameter of the tube in size, and blow up a small blister similar to that shown in Fig. 6.29a.

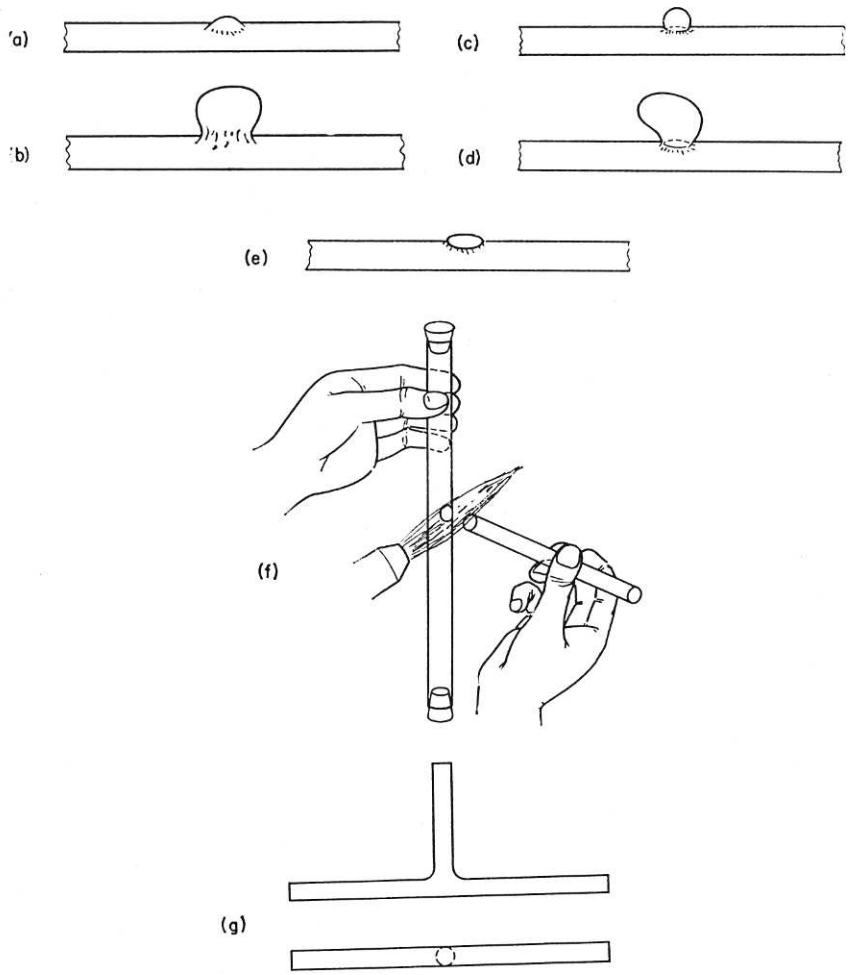

FIG. 6.29. Making a T-join. The diagrams should be studied when reading the text. Special attention should be given to blowing the hole in the cross-piece; if too much glass is heated and a large blister blown up (b), the T-join will be unnecessarily difficult to make.

The blister must be kept under close observation while it is being blown up. If blowing is overdone a shape such as that shown in Fig. 6.29b results, and difficulties of making a T-tube will be increased. The top of the blister is reheated and blown up as in Fig. 6.29c. The blister is again reheated and blown out (Fig. 6.29d).

The thin bulb is scraped off with the tweezers and the open end melted back and opened out with the small pointed tool so that it has the shape shown in Fig. 6.29e.

The hole must be circular and symmetrical with respect to the tube. It should be slightly smaller in diameter than the tube and of the same wall-thickness. Care in preparing the hole makes further work on the T-piece less troublesome.

The unfused end of the side-arm and the hole in the tube are both heated so that the edges are quite soft. They are then removed from the flame and immediately brought together so that there are no gaps or holes. The melted glass is at once pulled out by a very small amount and blown up very slightly. The side-arm is adjusted so that it is in the same plane as the cross-piece and at right angles to it. The join is now spotted with a small pointed flame, care being taken to avoid distorting the cross-piece or allowing the side-arm to be displaced in any way (Fig. 6.29g).

The T-piece is now annealed, with frequent close inspection and adjustments to keep the shape and angles correct. A number of T-pieces of various sizes should be made from 5 to 15 mm tubing. A very small sharp flame should be used for spotting so that melting is confined to the join only. Some care must be exercised to avoid blowing out the hot glass too much. The minimum amount of melting and blowing should be the aim.

Y-pieces

Making Y-pieces provides an opportunity to combine two techniques. Since the success of the second one, that is the join, depends very much on the quality of the first, that is the bend,

this exercise brings out the need for good wall-thickness on the outside of the bends.

Cut 10 pieces of 10 mm diameter tubing each 15 cm long and fire-polish both ends. Now cut 10 pieces of the same tubing each 8·0 cm long, and fire-polish one end. Bend the long pieces at the centre so that the angle between the limbs is 120°. Be sure that the worked glass is smooth, and that the wall is maintained at a thickness as near as possible to that of the tube. The angle between the limbs can be quite accurate if the final adjustment is made with the glass held over an asbestos sheet on which two limbs have previously been drawn. A hole is now blown on the bend so that its centre line is in the same plane as those of the bend limbs, and the diameter of the hole is 10 mm or a little less. The location, wall-thickness and diameter of this hole are of importance since they determine the symmetry and general neatness of the Y-piece (Fig. 6.30).

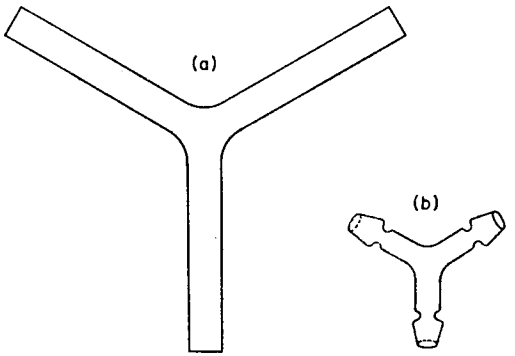

FIG 6.30. Y-pieces: (a) Shows the proportions and shape for practice work. A strong and compact form is shown in (b).

The open limb of the bend is stoppered and the short limb joined to the hole by the same procedure as that described for T-joins. All the worked glass is annealed and the limbs adjusted so that they are symmetrical with respect to one another, and all in the same plane. If the radius of the bend is made too small then

the glass on the outside will be very thin and difficult to rework, but if the radius is made too large then the finished Y-piece will not have a pleasing shape.

When experience is acquired in rotating the glass back and forth in the flame so that the join is uniformly melted all round, and the fingers of the left hand can be kept out of the flame, then both T and Y joins will be made more quickly and more neatly.

Joining Tubes of Different Diameter

1st Method. Pull double points on 18 mm tube with 5 cm between the shoulders. Cut one point open, pull the other point off and make a good test-tube end.

Adjust the air and gas to give a small pointed flame and melt the centre of the test-tube end. Blow out a small bulb, then reheat and open the bulb as in sketch. The hole should be slightly smaller than 8 mm diameter (Fig. 6.31c).

Now join a 15 cm length of 8 mm tube to the open end, confining all melting to the smaller tube and directing the flame so that the join is just melted. Keep everything straight and symmetrical and free from twists. The wall of the small tube should not be allowed to become excessively thick or thin. Very thin hot glass is difficult to control, while thick glass is difficult to bring back to the original wall-thickness without reworking. All such operations should be done quickly and neatly, with the minimum working of the glass necessary to make a good join (Fig. 6.31d).

2nd Method. Pull double points on 18 mm tube with 10 cm between the shoulders. Cut one point open. The points must be straight, strong and of adequate length, or the later steps will be made very difficult.

Make two small marks as in Fig. 6.32a. Use a flame of the same diameter as the tube. When the glass just softens pull out very slightly to give the shape shown in Fig. 6.32b. Continue heating with steady rotation till the hot glass constricts to look like Fig. 6.32c with uniform wall-thickness, no twists, and all in

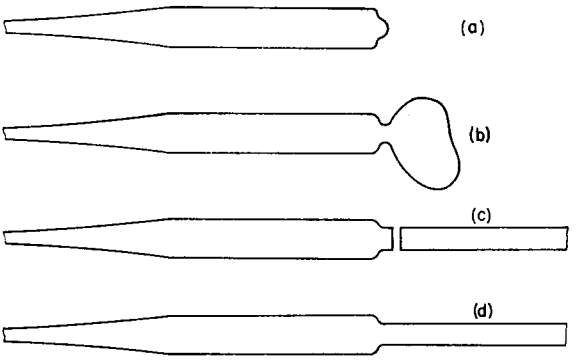

FIG. 6.31. A method of joining tubes of different diameter: (a) shows the test-tube end with the small bulb blown up. This bulb is blown out in (b). The completed join is shown in (d).

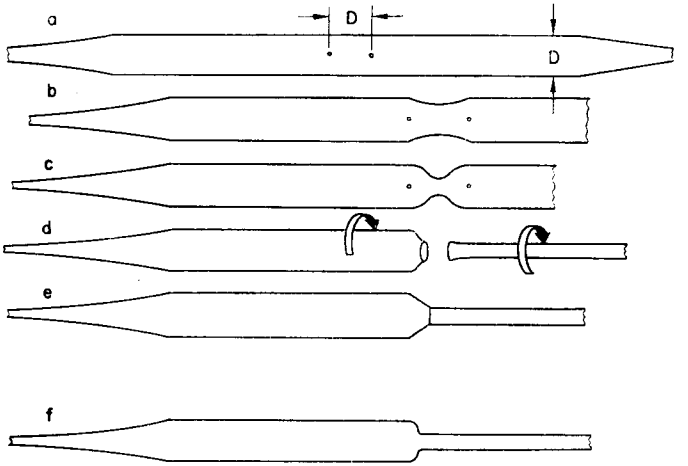

FIG. 6.32. Another method of joining tubes of different diameter: (a) shows the larger tube marked ready to be constricted; (b) the first step in making the constriction; (c) the finished constriction; (d) the tube ends ready to join together; (e) the shape of the join before blowing up; (f) the completed join.

line. The smallest diameter of the constriction should be just under 8 mm, i.e. 7–7·5 mm. Constricting tubes needs a great deal of practice and close attention to detail. The marks should appear on the newly formed shoulders as in Fig. 6.32c.

The size of the constriction must, of course, be judged by eye. This ability to judge sizes by inspection takes a little time to acquire, but comes with continued practice. When the glass has cooled, cut it in the middle of the constriction and close off the open point. There are now two 18 mm tubes ready to join to 8 mm tubes. In making the joins by this method the flame is directed on to the glass between the shoulder and the constriction, so that the smaller tube is melted as little as possible. When the join is thoroughly melted and the glass between it and the shoulder is all uniformly hot and slightly thickened, blow up slowly to the shape shown in Fig. 6.32f.

Although the second method appears to be more involved than the first, it will be found to be much quicker and to result in a neater shoulder and join.

Side Tubes and Water Tubes

Side tubes to be joined to test-tubes and U-tubes are cut off about 15 cm long, the end to be attached is opened out slightly with the small pointed tool, then annealed and set aside till required.

The test-tube is fitted with an asbestos stopper into which a 5 mm bore glass tube has been inserted, and marked about 4 cm down from the open end (Fig. 6.33).

After a preliminary warm up, a small hole the same diameter as the side tube is blown at the mark. The edge of the hole and that of the prepared side tube are heated until they are both quite soft, and the side tube end has shrunk in to the same diameter as the hole. They are quickly removed from the flame, gently pressed together so that there are no gaps and immediately pulled very slightly apart. The join is now remelted by rotating the test-tube about the centre line of the side tube. This calls for

concentration and practice. The side-arm must turn about its centre line, back and forth through at least 180°. The test-tube must be turned at the same time and through the same angle to

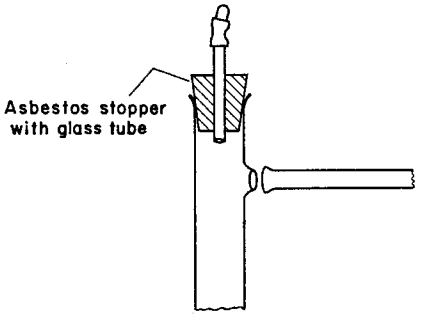

FIG. 6.33. Side tubes.

avoid twisting the melted glass. The left hand holding the test-tube must be kept out of the flame. The melted glass must not be stretched or otherwise distorted. When the join is well melted together it is blown out and pulled gently back into shape. The asbestos stopper is now removed and the test-tube annealed. Inspection and adjustment will be the same as that used for a T-piece. The side-arm is cut off to the required length and fire-polished. All the melting is done on the side tube and just enough of the test-tube is heated to allow the join to fuse together. Side tubes are joined to U-tubes in the same manner. The appearance will be spoiled if the side tubes are not in the same line (Fig. 6.34b).

When the side tube must be less than 3·5 cm from the open end of the test-tube, a different procedure is used. The steps are shown in Fig. 6.35. Should side tubes be required near the top of existing test-tubes the following method is recommended. The test-tube is marked with the knife at the place where the centre line of the side tube has to be. After a preliminary warm up, a very small sharp flame is used to heat the glass at the mark. The end of a suitable length of 3 mm diameter rod is attached to

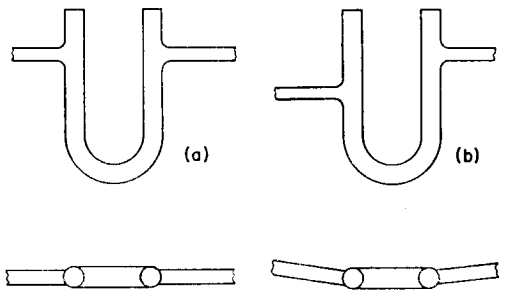

FIG. 6.34. Side tubes on a U-tube. (a) Shows a U-tube fitted with side tubes. (b) Shows the effect, on the appearance of the U-tube, of taking insufficient care when blowing out the hole in the second limb; the side tubes are not in line.

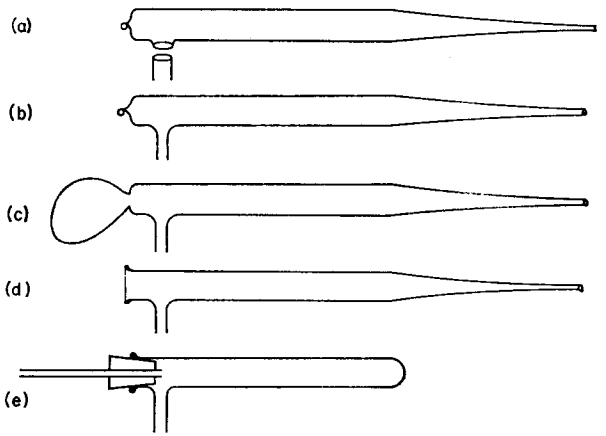

FIG. 6.35. Side tube near the end of a test-tube. (a) Double points are made from suitable tubing, one point is sealed off and the side tube attached as shown in (b). (c) The sealed off end is blown out and rimmed as in (d). (e) When the newly made rim has cooled, the open end of the test tube is fitted with a bored cork carrying a handle; the other end is sealed off and blown up to a neat hemisphere.

the heated spot and immediately drawn out (see Fig. 6.36a). This drawn-out glass is cut off near the test-tube wall, remelted and opened out until it is slightly larger than the side tube. The glass is carefully annealed and set aside.

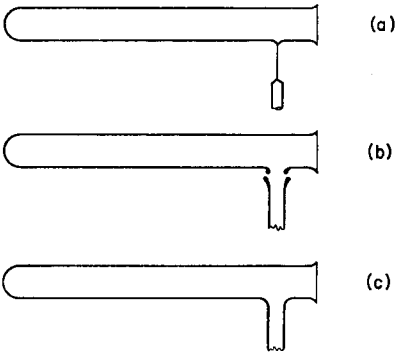

FIG. 6.36. Side tubes without blowing. (a) Shows the heated glass being drawn out with the rod. (b) The drawn out tube wall cut off and opened out to a hole, slightly larger than the bore of the side tube. The side tube end is shown reamed out and thickened. (c) The completed join made without blowing.

The side tube is prepared by softening about 1 cm of the end and opening out the bore to about $1\frac{1}{2}$ times its original diameter. The glass should be allowed to gather so that the wall of the flared tube is not less than that of the side tube. The hole in the test-tube and the prepared end of the side tube are both heated in a small flame until their edges are uniformly molten, but not melted in to a diameter less than that of the side tube. The edges are brought together, with no gaps, then gently pulled out. Although no blowing is possible with this method, it is quick, and, with a little practice and close attention to detail, strong joins of good appearance can be made. Careful and thorough annealing is necessary.

Bulb Blowing

A strong bulb of spherical shape, uniform wall-thickness and a predetermined diameter gives great satisfaction to its maker. With such an achievement behind him a glassblower can feel confident that he is making worthwhile progress.

To calculate the length of a tube that must be melted in order to blow a bulb of a given diameter:

Let us assume that a D mm diameter bulb is required in a d mm diameter tube, and that its wall-thickness must be very nearly the same as that of the tube.

Let t be the wall-thickness of the tube.

Let d be the diameter of the tube, and l be the length of the tube required to make the bulb.

Let D be the diameter of the bulb of wall-thickness t and all dimensions being measured in millimetres.

Then the volume of glass melted will be πdlt approximately, and the volume of glass in the bulb $\pi D^2 t$ approximately and

$$\pi dlt = \pi D^2 t \text{ mm}^3$$

and

$$dl = D^2,$$

therefore

$$l = D^2/d \text{ mm}.$$

The length of tube to be melted = (bulb diameter)^2/the tube diameter.

Spherical bulbs are very strong, both mechanically and thermally, and the wall-thicknees need not be as great as that of the attached tubes. Also in the above reasoning no account has been taken of the fact that the bulb is not quite a complete sphere, since it will have two tubes attached to it. We can make a reasonable allowance for these imperfections by taking

$$l = \frac{D^2}{d} - \frac{d}{2}$$

for bulbs of the same wall-thickness as the tube,

and $$l = \frac{4}{5}\left(\frac{D^2}{d} - \frac{d}{2}\right)$$

for bulbs of slightly thinner wall than the tube.

To blow a 10 mm diameter bulb in a 5 mm diameter tube, cut off a 30 cm length of the tube and fire-polish the ends. Make two marks near the centre and 16 mm apart. Stopper one end with a rubber plug.

Hold the glass in the manner described for pulling points and heat and gather all the tube between the marks, so that the wall-thickness is approximately doubled while the bore is only very slightly constricted (see Fig. 6.37a).

Now bring the open end of the tube, in the right hand, up to the mouth and blow the molten glass up into a bulb. Steady rotation must not be stopped, the tube must be kept in line and the molten glass must not be stretched or twisted. The bulb is inspected before the glass sets, and pulled or pushed very gently to give a spherical shape. The flame size should be adjusted so that it will heat a length of the tube equal to twice its diameter. Very little air is required. The tube must be turned uniformly

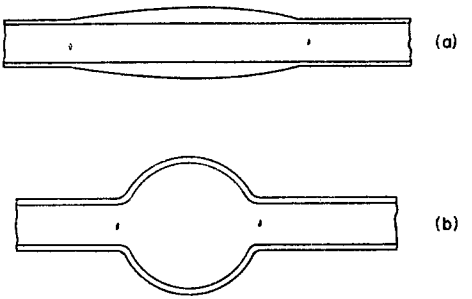

FIG. 6.37. Bulb blowing. (a) The length of glass required for the bulb is marked on the selected tube. All the glass between these marks is heated and gathered, as shown, before the bulb is blown. (b) Shows the bulb blown to the required size, and the final position of the marks.

throughout the whole operation; it must be kept steady and in the flame, or the bulb will blow up asymmetrically. Considerable care should be exercised in judging, by eye, when the bulb is 10 mm in diameter. Most beginners blow the glass before enough has been melted, and also blow it up too far (see Fig. 6.37b).

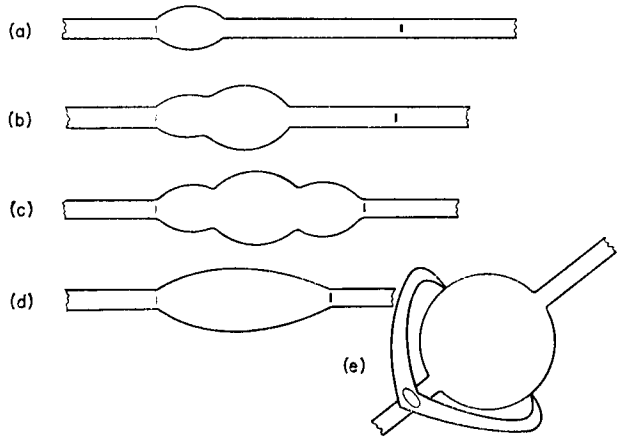

FIG. 6.38. Large bulbs. The glass is gathered up by blowing a succession of small bulbs as shown in (a), (b) and (c). The wall-thickness of these small bulbs should not be less than that of the tube. (d) All the glass between the marks is then gathered in to a smooth mass of nearly uniform wall-thickness and free from constrictions. (e) The bulb is blown up and measured with calipers. Some care is necessary to avoid making the bulb too large.

This exercise should be repeated till good bulbs of up to 3 times the diameter of the tube can be blown in all sizes up to 15 mm diameter. If the bulb diameter has to be more than about 3 times that of the tube, or if the gas supply is insufficient to melt all the glass required at one heating, then the following procedure is recommended.

Blow a series of bulbs, of good shape and wall-thickness, close together, until all the necessary glass has been melted. The air-flow should be just enough to soften and gather the glass (Fig. 6.38). All the worked glass is now heated and gathered

into a smooth mass, free from twists or constrictions, and blown up to the required size with steadily increasing air pressure.

Some care must be taken to avoid blowing too large a bulb; although it is fairly easy to reheat a bulb and blow it up slightly, it is much more difficult to re-melt and re-blow to a smaller diameter. It is not easy to judge bulb diameters by eye. A good plan is to use the tube diameter as a unit, and to remember the ratio of the bulb diameter to the tube diameter.

To blow a bulb on the end of a tube

First close the tube by drawing out a point and making a neat hemispherical test-tube end. Then heat the glass, including the test-tube end until sufficient glass has been gathered. The tube should have the appearance shown in Fig. 6.39a.

In this case

$$l = \frac{D^2}{d} - \frac{d}{4}.$$

Since in this case the tube must be held in one hand only, rather more difficulty will be experienced in gathering the glass. The tube should therefore be held in the flame at an angle below the horizontal. This angle is usually about 45° but must be varied as required, to keep the molten glass in line with the tube, and uniformly thick. Be particularly careful to have the test-tube end uniformly hot but not much thicker than the rest of the gathered glass. Some care must be exercised to prevent the hot glass from sagging down to one side. As soon as such a tendency is noticed the tube must be turned so that the sagging glass falls back into line. The bulb is blown up with gradually increasing pressure and steady rotation, until it reaches the required diameter.

If the bulb has to undergo further working, e.g. if a side tube is to be attached as in Fig. 6.39b, then it is essential to melt and gather sufficient glass to make a bulb of wall-thickness very near to that of the tube, 1–1½ mm. Bulbs with a wall-thickness

of $\frac{1}{2}$ mm or less are very difficult to rework. Thin glass heats up and collapses very rapidly, and attempts to recover the smooth round shape may prove impossible.

Should the bulb diameter be very much greater than the attached tubes it is better to blow it from glass of nearly the same diameter as the bulb. Two methods will now be described.

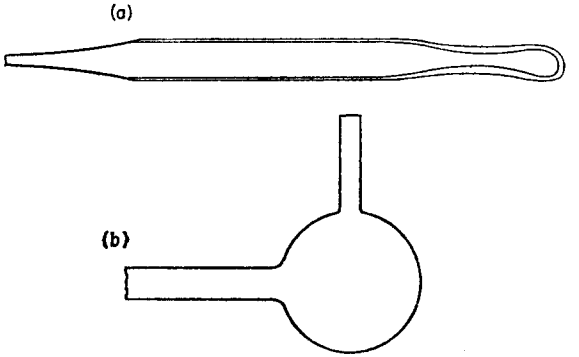

FIG. 6.39. Blowing a bulb on the end of a tube. First prepare a test-tube end on a suitable piece of tubing and mark the required length on the tube. (a) Shows the glass heated and gathered, ready to blow up. (b) The blown bulb with a side tube attached. Such a shape is difficult to preserve if the bulb is thin-walled.

From a tube whose diameter is approximately four-fifths that of the required bulb, draw strong double points with the distance between the shoulders equal to the bulb diameter (see Fig. 6.40a).

A constriction is now made at each shoulder as shown in Fig. 6.40b. These constrictions must be reasonably thick and strong—the virtues of substantial points will now be apparent. All the glass between these constrictions is now heated in a flame adjusted so that it is just hot enough to gather the glass. The bulb is now blown up with steady, continued rotation and carefully modulated pressure. The tubes are now joined to the constrictions without disturbing the bulb.

In the other method, the tubes are joined to the constrictions before the bulb is blown. This method makes a neater job, but

extra care must be exercised to avoid melting the relatively small diameter tubes when the glass for the bulb is being heated and gathered (Fig. 6.40e,f).

When bulbs of more than 50 ml capacity have to be incorporated in apparatus it is much easier to use round-bottomed flasks, obtained from the laboratory store. These bulbs must be made from the same type of glass as the tube stock, or there will be a considerable risk that the joins will crack on cooling.

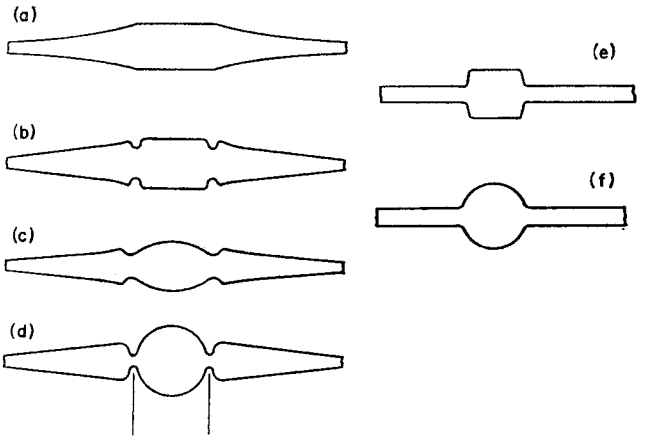

FIG. 6.40. Blowing a bulb from a large diameter tube. (a) Shows double points drawn from tubing of four-fifths the diameter of the required bulb. (b) The constrictions made at the shoulders. (c) The heated and gathered glass ready for blowing up to the desired size, as shown in (d). (e) Shows tubes joined to the constrictions before blowing the bulb. (f) The finished bulb with tubes attached.

Riffled Side Tubes or Water Tubes

When side tubes are required with riffles or corrugations so that attached rubber tube will remain securely in place, then these tubes are prepared before they are joined to the glassware.

It is advisable to have a number of these on hand; they are prepared as follows:

Pull strong double points, with 10 cm between the shoulders, in 8–9 mm normal walled tube. Open one point on each, make a constriction at the shoulder, to about 6 mm o.d. and of the same wall-thickness as the tube. Then make another similar constric-

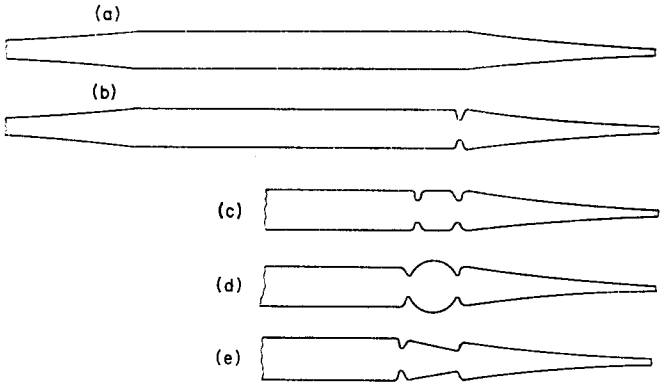

FIG. 6.41. Riffled side tubes. These tubes are prepared in pairs from double points shown in (a). (b) The first constriction and (c) the second constriction must be neat and of reasonable wall-thickness. (d) Shows the bulb blown up. (e) Shows the bulb drawn into shape after heating on one side. The procedure is repeated at the other end of the double point.

tion 1 cm along. Allow the glass to cool slightly. Now heat the glass between these constrictions and blow it into a small bulb and again allow the glass to cool slightly. Reheat this bulb on the side nearest to the open point and gently pull it out to the shape shown in Fig. 6.41 e. Flame-anneal and set up to cool. The point must not be cut off at this stage as it is necessary as a holder when the side tube is being attached to glassware in hand. When both ends of all the double points have been shaped as described, they are set aside until required.

Ring Seals, Multiple Seals or Internal Seals

When skill and competence have been acquired in joining tubes of the same, and different, sizes, and in making smooth bends and neat bulbs, then it is desirable to practise making ring seals. These are of two types.

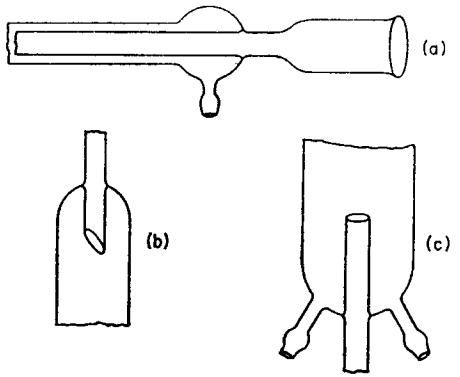

FIG. 6.42. Internal seals. (a) Internal seal in a Liebig condenser. (b) A drop counter, and (c) a constant level device.

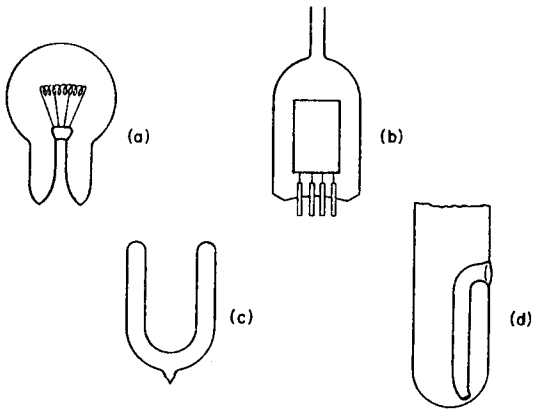

FIG. 6.43. Ring seals in (a) an electric lamp, (b) a radio valve, (c) a Dewar flask, and (d) part of a steam distillation apparatus.

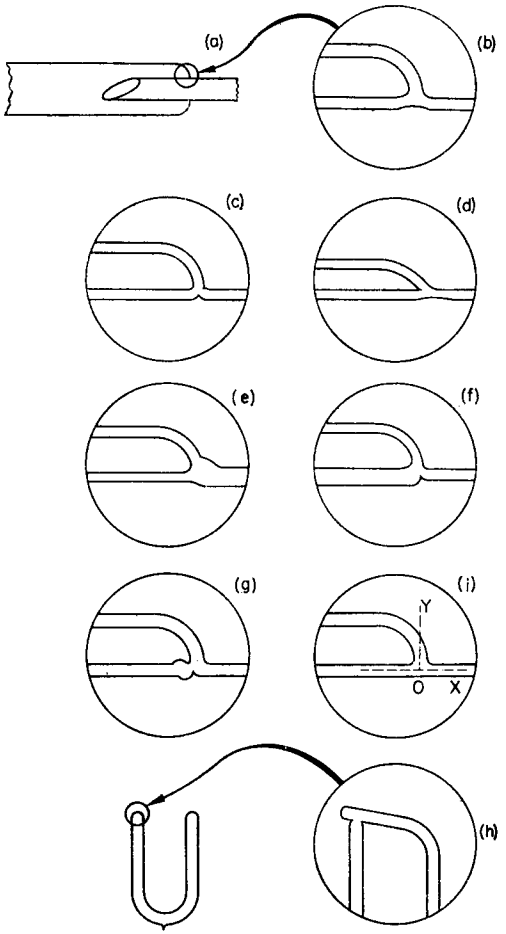

FIG. 6.44. Some faults in internal seals and ring seals. (a) A ring seal in a drop counter. (b) Shows a desirable shape. (c) Re-entrant angles resulting from insufficient melting. (d) Re-entrant angles due to faulty shape. (e) Excessive variation in wall-thickness. (f) Internal tube with greater wall-thickness than outer tube. (g) and (h) Re-entrant angles, insufficient melting. (i) Shows a desirable shape for an internal seal. The wall of the outer tube is slightly greater than that of the inner tube; the joins are smoothly rounded; the angle *YOX* is very nearly a right angle.

In the first type (Fig. 6.42) one glass tube passes through the wall of another, as in condensers, drop counters and constant level devices.

The second is fairly common and seen in Dewar flasks, electric lamps and radio valves. One tube is inserted into another of larger diameter and their open ends are joined together (Fig. 6.43).

For such seals to survive and give useful service they must be of good quality, that is, there must be no great variation in wall-thickness, and the joins must be thoroughly melted together and free from re-entrant angles.

In Fig. 6.44b is an enlargement of Fig. 6.44a and shows the detail of a good type of internal seal. Figure 6.44c–h show faults to be avoided, such faults may cause the join to crack, sooner or later, no matter how carefully the glassware is annealed. The most dangerous faults are: large variations in wall-thickness in the vicinity of the ring seal, and re-entrant angles, which are almost always the result of insufficient melting. In Fig. 6.44i the angle *YOX* should be as nearly a right angle as possible to reduce the chance of re-entrant angles being left on the internal surface of the seal.

A Saliva Trap

The first steps required in making a saliva trap will be clear from Fig. 6.45. The ring seal is made by inserting the small tube into the large bulb, then melting the small bulb with a sharp hot flame so that it joins to the thin edged hole. Heating is confined to the small bulb and just enough of the large bulb melted to give a good join. The glass must be very hot so that it flows together to give smoothly curved surfaces. Close attention should be paid to wall-thickness and tube diameter, to keep them uniform. Everything must be kept in line; the short inner tube will flop about, but can be kept reasonably central with careful manipulation of the glass so that it falls always towards the centre line.

When the ring seal is satisfactorily completed the hot and

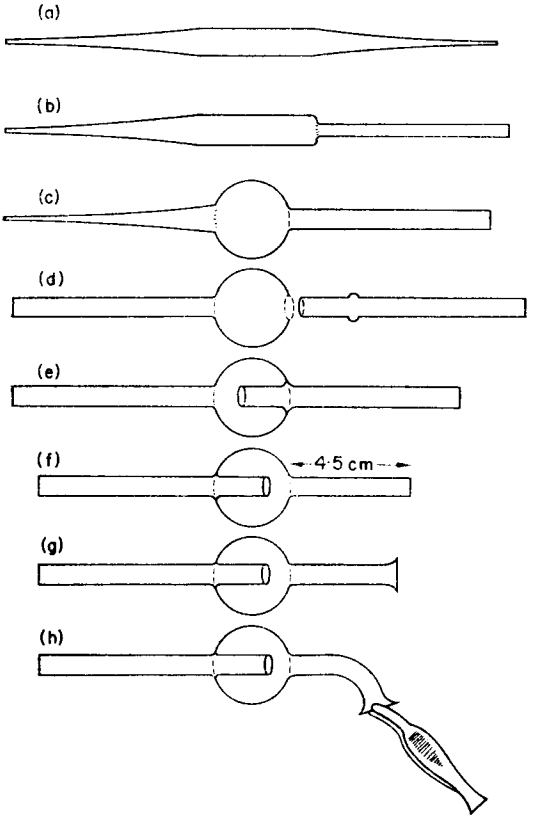

FIG. 6.45. A saliva trap. (a) Double points made from 15 mm tubing. 3 cm between the shoulders. (b) A 15 cm length of 6 mm diameter tube joined on. (c) All the 15 mm tube used to blow the bulb. (d) The bulb is reversed in the hands and the end blown out to leave a neat hole. A small bulb is blown in another piece of 6 mm tubing, one end is cut off as shown. (e) The small bulb is sealed to the hole in the large bulb. (f) The trap is again reversed and the right hand tube cut off 4·5 cm long. (g) The end of this tube is flared. (h) Shows the mouthpiece being bent, with tweezers, after heating in a soft flame.

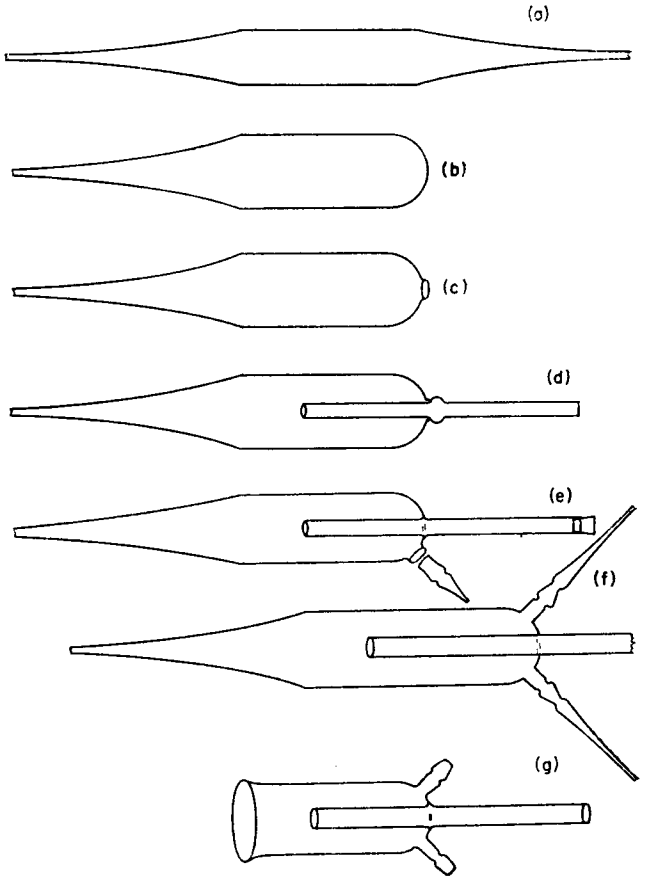

FIG. 6.46. A constant level device. (a) Double points from 25 mm diameter tubing. (b) One end blown to a hemisphere. (c) Shows a 8 mm diameter hole blown in the hemispherical end. (d) The prepared overflow tube, of 6 mm tube and with a 10 mm diameter bulb, inserted into the body of the constant level device. (e) The ring seal completed, and the inlet tube ready to join on. (f) The outlet tube joined on. All the heated glass, at this end of the device, should be annealed. (g) Shows the completed constant level device with the top blown out and rimmed.

plastic small bulb can be blown and pulled back to the original diameter and wall-thickness. The saliva trap is now flame-annealed, and set up in the rack to cool. The trap is finished by cutting off the top tube 4·5 cm from the bulb and flanging out the end; then, using a large soft flame, the tube is softened and carefully bent with the tweezers to the shape shown in Fig. 6.45h. After local flame-annealing of the parts touched and cooled by the tweezers, the other end is cut off 4·0 cm from the ring seal and fire-polished.

A Constant Level Device

The successive steps taken and the dimensions used in making a constant level device are shown in Fig. 6.46. All the parts must be prepared before the assembly is undertaken. The ring seal is completed then *kept hot* till both side tubes have been attached.

The glass surrounding the ring seal and water inlet and outlet tubes is carefully flame-annealed, care being taken to centre the inner tube with respect to the reservoir.

The top of the device is then blown out and finished with a slightly thickened rim. When the glass has cooled the inlet and outlet tubes and the overflow tube are cut to length and fire-polished.

A Simple All-glass Condenser

The five parts of the condenser are prepared first. The jacket and vapour inlet are made together from a 20 mm diameter tube 18 cm long with a rimmed and slightly tapered open end. The other end is closed and blown out to a neat hemisphere of uniform wall-thickness. The inner tube has two straight cut ends, one flanged out so that it just enters the jacket, the other melted and opened out very slightly.

The liquid drainage tube is a 15 cm length of 10 mm tube with one end fire-polished. The water jacket inlet and outlet tubes are taken from the prepared stock, separated by cutting with the glass knife; the open ends are slightly enlarged and

thickened as already described on page 102. An asbestos stopper, with a close fitting glass tube, is used to hold and rotate the condenser body and vapour inlet tube (Fig. 6.47).

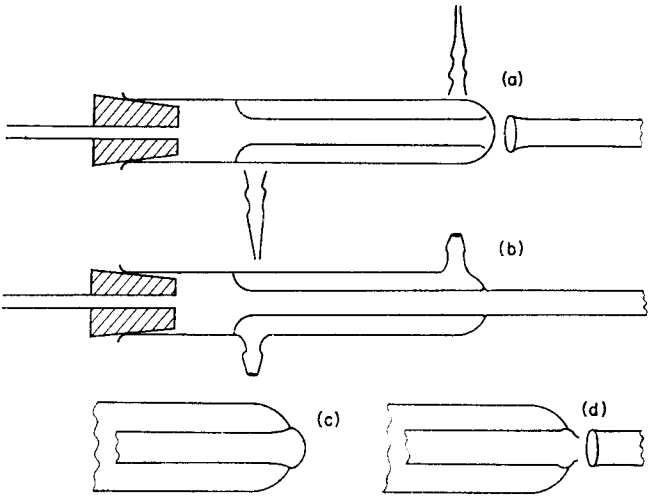

FIG. 6.47. A simple condenser. (a) Shows the proportions of the five parts. The large test tube is 18 cm long and 20 mm in diameter. (b) The completed condenser. The order of steps taken in the assembly of the parts is described in the text. (c) and (d) Details of the formation of the first internal seal.

The prepared inner tube is placed in the jacket, with the small end in contact with the test-tube end. The stopper is inserted in the jacket and the ring seal made. The inner tube will be kept reasonably centred at one end by the close-fitting flange. The other end can be kept central by tilting the jacket to about 60° above the horizontal so that the small end of the inner tube is supported by the hemispherical inside wall of the test-tube end. After careful preliminary heating the ring seal is made with a fairly small hot flame. Heating and melting is confined to the ring seal and should not be allowed to spread to the shoulder. The inclination of the jacket must be adjusted to preserve the

shape of the hemispherical end. The seal is satisfactory when it appears to have a slightly greater wall-thickness than that of the inner tube. The flame size is now reduced and the glass disc within the ring seal is heated and blown up into a neat uniform hemisphere. This hemisphere is blown out and joined to the liquid drainage tube. The water inlet tube is now attached to the jacket. The ring seal must be kept hot during this operation.

The ring seal and all the glass surrounding the water inlet tube are now carefully flame-annealed, and the condenser is allowed to cool. If oven-annealing is inconvenient, or impossible when the condenser is completed, then at least 2 hr (and preferably 24 hr) should elapse before the second ring seal is completed. Glass takes some time to completely contract on cooling from a high temperature. Contraction lags behind cooling. Since this condenser has two ring seals, and since the inner tube will cool rather more slowly and through a somewhat shorter temperature range than the jacket, it is important that the residual strain set up be kept to a minimum. This condition will be more nearly approached if the residual strain is that due to cooling one end only.

The condenser is now held with the liquid drainage tube in the left hand. The second ring seal is made with very thorough melting followed by blowing to recover the original wall-thickness and tube diameter. The first water tube is used to blow up the water jacket and to blow out the hole for the second water tube. The water outlet tube is now attached to the jacket and as near to the ring seal as possible. This operation must be carried out without delay, or the ring seal must be reheated occasionally to prevent it from cracking.

The flame-annealing technique employed with this second end is important. It should be studied, understood and applied whenever this type of glassware is made.

All the glass surrounding the ring seal and the water outlet tube is annealed in the normal manner. The flame is then reduced in size and the air-flow adjusted so that the flame just loses its yellow colour. This flame is now directed on to the rotating vapour inlet tube so that the ring seal is kept hot but the glass

of the condenser body is not in the flame. This heating should continue for some time to allow the condenser jacket and inner tube to cool and to ensure that at least some of the differential contraction is taken up by the still hot ring seal.

References

1. THRELFAL, R., *On Laboratory Arts*, Macmillan, 1898.
2. WM. STEVENSON, Glasgow, the author's first tutor.

CHAPTER 7

Glass-to-Metal Seals

PLATINUM wire of small diameter has been sealed through the walls of soft glass apparatus for many years. These wires are used as a conducting path for electric current, and the seals are vacuum tight.

The development and extensive application of glasses other than lead and soda has resulted in much research into the properties of metals and their suitability for glass to metal seals. As a result vacuum tight seals of sufficient mechanical and thermal strength for normal use, and varying from tiny wires carrying current to micro lamps, to heavy tungsten rods in mercury arc rectifiers, and large metal flanges found in the base of radio transmitting valves, are now made in large numbers.

The manufacture of many of these seals requires specialized materials, equipment and techniques that are only possible with large-scale production. A limited number can be made in the glassblower's workshop with simple materials and equipment.

Partridge[1] has assigned glass-to-metal seals to four types:

(1) "Matched" seals in which the metal is sealed directly to the glass, the resulting stress in which is kept within a safe limit by selecting a glass and a metal whose coefficients of thermal expansion and contraction are closely alike.

(2) "Unmatched" seals in which the thermal expansion of the metal differs from that of the glass, and the dangerously high stresses which would normally arise are avoided by using (a) metal of small diameter; (b) ductile metals which, by their yielding, give some relief to the stresses in the glass; or (c) intermediate glasses and graded seals; the final

seal between the metal and the last member of the intermediate glasses being of the "matched" type.

(3) Soldered seals in which the metal member is soldered to a layer of metal previously applied to the surface of the glass by one of several convenient methods.

(4) Mechanical joints or seals, between glass and metal.

The selection of suitable glasses and metals, or alloys, their preparation and sealing are fully dealt with by Partridge.[1] He lists the following conditions to be met by the metal component:

(1) Its melting point must be higher than the working temperature of the glass.

(2) Sufficient quantities of it, of specified thermal expansion coefficient, should be available in a clean state from the metallurgical point of view, i.e. as free as possible from non-metallic inclusions.

(3) It must be sufficiently ductile to enable it to be formed into wire or strip without cracks, seams, laps or other mechanical defects.

(4) The curves of thermal expansion vs. temperature of both metal and glass should, in the case of matched seals, follow one another closely over the same specified range of temperature.

(5) No allotropic transformations, accompanied by marked changes in expansion rate, should occur in the metal over the range of temperature to which it may be subjected, either in making the seal or during its subsequent use. This range may be as extensive as $-50\,^{\circ}C$ to $2000\,^{\circ}C$.

(6) Any layer of oxide formed during the process of making the glass-to-metal seal should adhere firmly to both metal and glass.

(7) High electrical and thermal conductivity are advantageous if the metal has to carry a substantial electric current, for otherwise the heating effects when the current flows may result in a substantial increase in strain.

Sealing Platinum Wire in Soda Glass

Platinum wire of suitable length and not more than about 0·8 mm diameter is held by clean forceps in a small oxidizing flame so that one end becomes bright red. At the same time one end of a 10 cm length of 5 mm diameter lead glass rod is melted in the flame. The wire is then pushed into the hot glass; and the rod is then used as a holder (Fig. 7.1a).

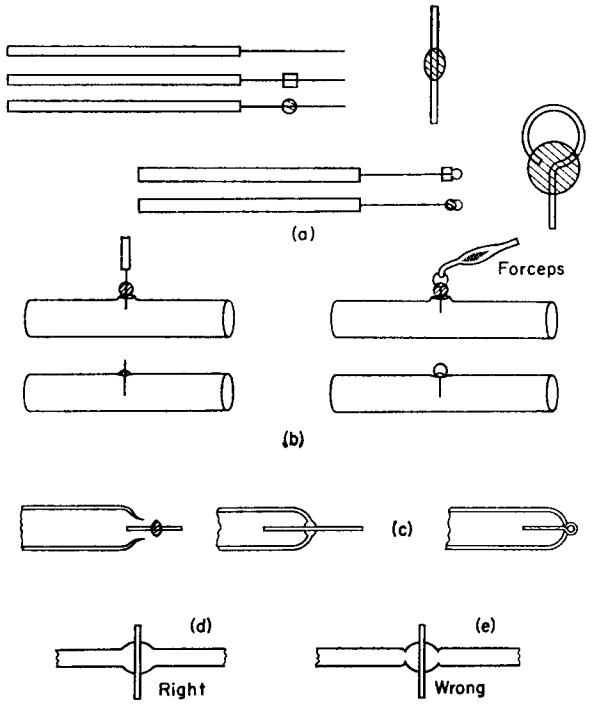

FIG. 7.1. (a) Beading a platinum wire into lead glass. The wire may be straight or bent into a circle. (b) Sealing the beaded wire into a hole in the side of a glass tube. (c) Sealing a beaded wire into the end of a glass tube. (d) An enlarged view of the glass wall showing no great variation in wall-thickness in the vicinity of the platinum wire. (e) An enlarged view showing variations in wall thickness likely to cause cracks.

A 3 mm length of lead glass tube of small diameter is threaded on to the wire; and melted into a sphere in the oxidizing flame. The wire must be rotated to prevent the glass bead from flowing to one side and becoming eccentric. Melting should proceed from one end of the tube to avoid trapping air bubbles between the metal and the glass. The lead glass must be worked in the tip of the oxidizing flame to avoid blackening its surface.

The bead of lead glass is now sealed into a suitable hole blown in the wall of the soda glass tube, using the same oxidizing flame. The bead must be thoroughly melted so that the junction between the lead and soda glass is smooth and free from re-entrant angles. Such re-entrant angles weaken the seal and may cause it to crack. The lead glass surface must be kept free from reduced lead throughout the sealing operation, but should reduced lead appear on the outside surface of the bead during flame annealing it will do no harm, as the seal between the platinum and the lead glass and the join between the lead glass and the soda glass are now completed (Fig. 7.1 d).

This type of seal is vacuum tight and very common in laboratory apparatus.

Sealing Platinum Wire in Borosilicate Glass

Thin platinum wire of about 0·25 mm diameter may be sealed in borosilicate glass. The seal is unlikely to be vacuum tight, or liquid tight, but will provide a useful electric contact through the glass to mercury within the apparatus. Thin platinum melts in the type of flame used for working borosilicate glass and a different technique must be used.

A short length, about 5 mm, of the wire is hammered to about 0·01 mm thick and the wire is sealed into a constriction previously made in a tube. The wire is not exposed to the oxy-coal-gas flame (see Fig. 7.2).

Platinum is a very expensive metal and therefore unsuitable for such mass-produced glass-to-metal seals as are found in radio valves and electric lamps. No other pure metal has a

suitable coefficient of expansion or a suitable electrical resistance for sealing to lead or soda glasses. A composite wire has been developed as a substitute for platinum. It consists of a core of nickel–iron alloy (43% nickel) covered with a thin sheath of

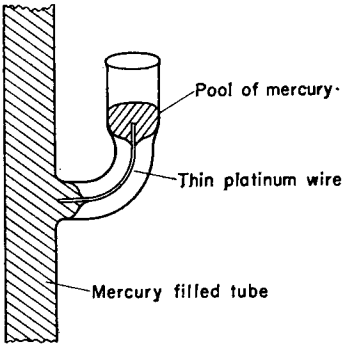

FIG. 7.2. A thin platinum wire sealed into a borosilicate tube to make contact with mercury at both ends of the wire.

electrically deposited copper. The composite wire is drawn out to about 0·5 mm diameter, the copper sheath is then about 0·03 mm thick. This composite wire has a comparatively high electrical resistance and a short length, just sufficient for the seal, is butt welded to a nickel wire on the vacuum side of the seal and to a copper wire on the other side.

The thermal expansion of Dumet, as this composite wire is called, is about $7{\cdot}0 \times 10^{-6}$ per °C in the longitudinal direction of the wire, and in the radial direction it is about $9{\cdot}1 \times 10^{-6}$ per °C which is nearly the same as that of lead glass. Seals made between Dumet and lead glass are only partly annealed and the glass is left in compression in the longitudinal direction.

Electric lamp and radio valve pinch seals usually have two or more wire leads passing through the glass. A simple form is shown in Fig. 7.3.

The wires can be purchased[2] in a number of sizes, ready to seal into the glass.

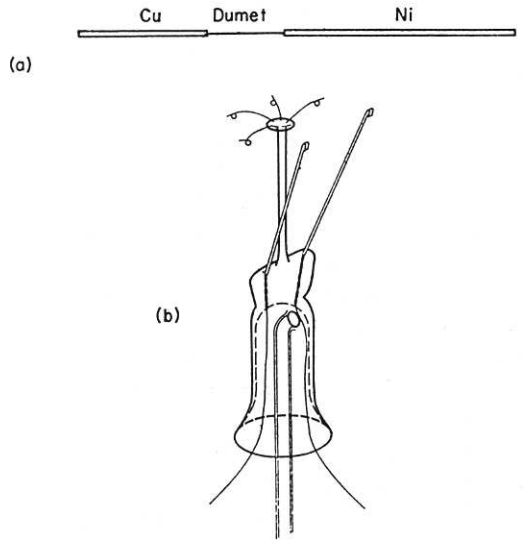

FIG. 7.3. (a) Shows a prepared Dumet wire. One end is welded to a length of copper wire, the other end is welded to a nickel wire. The copper sheath of the Dumet is borated. (b) Dumet wires sealed into an electric lamp foot.

Glass does not adhere readily to copper or its oxide and the copper surface should be "borated". Borated copper has a bright red colour due to a coating of fused borax and copper oxide.

Sealing Tungsten Wire to Borosilicate Glass

Any good quality tungsten wire of about 1 mm diameter can be used for practice exercises. If, however, the seal is to be used in some important equipment, then specially prepared wire, known as centreless ground tungsten rod,[2] should be used. This rod is clean, and free from surface defects which can result in leaks into vacuum apparatus that are difficult to detect.

The preparation of the tungsten. A number of methods for cleaning the surface of tungsten wire are described by Partridge.[1]

It has been found that centreless ground tungsten rod[2] is clean and, provided the surface to be incorporated in the seal is kept free from contamination and finger-marks, it can be used without further preparation.

The wire is cut to length with the glass-cutting carborundum wheel described in Chapter 4. If pliers or wire-cutters are used the wire is liable to split and be rendered unusable. Centreless ground rod can be purchased in 10 cm lengths or the length can be specified when the order is placed.

The selected wire is held in a pin-vice, or temporarily joined to a borosilicate rod handle, as a preliminary to cleaning. The centre of the wire is heated to dull redness and immediately rubbed with sodium nitrite. The sodium nitrite combines with the oxide and with the tungsten metal to leave a clean metallic surface on the wire. This treatment should not be prolonged, or the diameter of the wire will be reduced; the reaction is exothermic, generating sufficient heat to keep the metal red hot, and it will continue if the hot wire is kept in contact with sodium nitrite.

The cleaned wire is washed in tap water to remove the sodium nitrite and rinsed in distilled water.

Since hot glass will adhere to tungsten oxide but not to the metal itself, and since part of the oxide layer dissolves in the glass, it is important that this layer be of suitable thickness; if too thick, the oxide layer may separate from the metal surface, and if too thin it may all dissolve in the glass. The clean tungsten wire is gently heated in an oxidizing flame till the metallic surface shows a blue–green colour, then heating is stopped. A previously prepared borosilicate glass tube 2 cm long, about 1·5 mm in wall-thickness, and an easy sliding fit, is slipped on to the wire and moved along to its centre. Some care must be taken to avoid damaging the oxide layer. This tube is melted on to the wire with a small hot pointed flame. Melting must proceed from one end, with continued rotation, to prevent air being trapped between the glass and the wire. When the sealing is complete, the wire, as seen through the glass sleeve, will be magnified. A flat button of borosilicate glass, between 6 and 8 mm in diameter, is now built up in the centre of the glass sleeve by winding on a 1 mm dia-

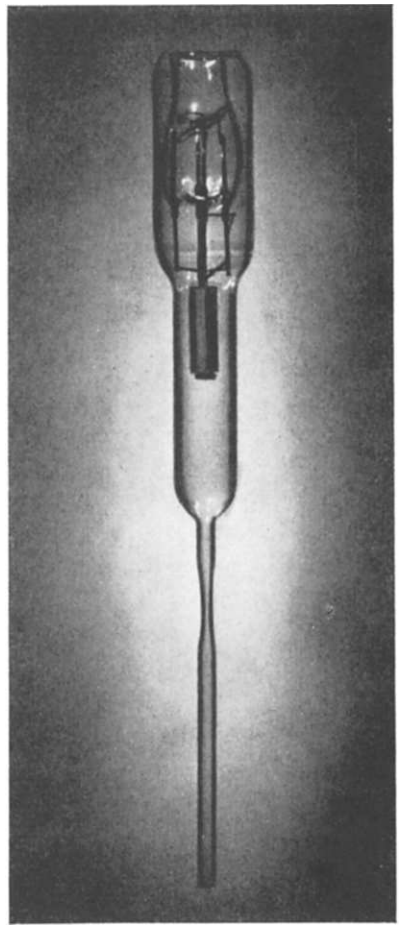

PLATE 7.1. An experimental gas discharge tube electrode. Tungsten wires sealed through Pyrex glass.

meter rod. Care must be taken to ensure that successive layers of the glass rod are thoroughly melted together as any pin holes that may be left will be difficult, if not impossible, to correct at a later stage (Fig. 7.4).

The interface between the borosilicate glass and the tungsten metal should be pale yellow to light brown when the seal has cooled. Should the colour be that of the clean metal the oxide

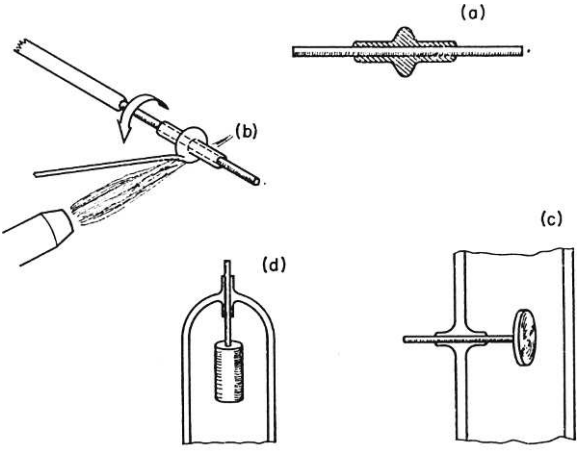

FIG. 7.4. Seals made with tungsten wire and borosilicate glass. (a) A prepared wire with sleeves and button. (b) Building up the button with thin rod. (c) and (d) show two electrodes with this type of seal.

layer is too thin. Should it be dark brown or black, the layer is too thick. In either case the seal will leak under vacuum and be unsatisfactory.

Electrodes and leads can be brazed or silver soldered to the clean tungsten wire; or, if facilities are available, they can be spotwelded to the tungsten. The seal is joined to the glass apparatus by blowing a hole in the glass wall of the same diameter as the button and melting them together. A smooth join with no excessively thick regions and no re-entrant angles should be the objective.

If wires of more than 1 mm must be used then it is advisable to make the sleeve from a tungsten sealing glass such as

Chance Bros.[3]	GS 1
G.E.C.[4]	W 1
C.T.C.[5]	B 37

The button can be made from the same borosilicate glass as the remainder of the apparatus.

Joining Glasses of Different Types

Glasses can be joined together successfully if the difference in their coefficients of expansion is small. In practice this difference may be as great as $1{\cdot}5 \times 10^{-6}$ per °C, but a difference of 1×10^{-6} per °C, or less, is to be preferred.

Should it be necessary to join two glasses together whose expansion coefficients differ by more than $1{\cdot}5 \times 10^{-6}$ per °C, a

TABLE 4. GRADED SEAL GLASSES

Code reference	Expansion coefficient $\times 10^{-6}$ per °C	Annealing temperature °C
Silica	0·5	1050
GS 10	1·0	750
GS 20	2·0	730
GS 25	2·5	690
GS 30	3·0	640
Pyrex	3·2	560
B 37	3·75	525
GS 44	4·25	550
GS 50	4·9	520
GS 65	6·45	535
GS 77	7·5	550
GS 85	8·3	540
S 96	9·2	530

number of intermediate glasses must be used. Such a series of joins is known as a graded seal and is used to seal metals to otherwise unsuitable glasses, or to join silica tubes to borosilicate or soda glass tubes.

Unless graded seals are required in reasonably large numbers, or for unusual purposes, it is more economical to buy them ready-made, rather than to maintain a stock of the intermediate glasses. These glasses have limited application and are expensive.

Table 4 gives graded seal glasses and is taken from the makers'[5] catalogues.

It is clear that oven annealing will be ineffective and that each join in a graded seal will have some residual thermal strain. This can be slightly reduced by flame annealing the joins as they are completed.

References

1. PARTRIDGE, J. H., *Glass to Metal Seals*, Society of Glass Technology, 1949.
2. Jencons Scientific Ltd., Mark Road, Hemel Hempstead, Hertfordshire, England.
3. and 4. Although these glasses are no longer in production, some stock may be available.
5. Glass Tubes and Components Ltd., Sheffield Road, Chesterfield, Derbyshire, England.

CHAPTER 8

Intermediate Glassblowing

MANY items of laboratory glassware can be made and repaired when reasonable competence has been acquired in the basic operations described in Chapter 6, and flame annealing is properly understood and can be effectively used.

Although it is commonly thought that it is easier to repair broken glassware than to remake the item, this is not always true. In many cases repairs can be undertaken only by those who are capable of making the items in question.

Repairs

Simple glassware, such as test-tubes, T- and Y-pieces, wash-bottle parts and glass pieces for joining rubber tubing should be renewed rather than repaired. Indeed, all glassware brought for repair should be examined carefully and a decision made whether a repair or a renewal will take least time and be equally satisfactory. For example, if the water jacket of an all-glass Liebig condenser is broken then a new condenser should be made; should the condenser drainage tube or vapour inlet tube or water inlet or water outlet be broken, they can be replaced. If the water jacket of a spiral coil condenser is broken, an attempt to salvage the glass spiral and to incorporate it in a new condenser will be justified.

Bulb and single graduation pipettes should be discarded if the jet is damaged, or if the break occurs between the graduation mark and the jet. The only acceptable repair is one that does not affect the capacity of the pipette.

Graduated pipettes may have the tip repaired by drawing out the glass to the desired bore, and cutting and grinding it to a new jet. A glass-cutting machine with a rubber-bonded carborundum wheel can be used to do this type of grinding. A light pressure of the glass on the side of the wheel, with steady rotation in the stream of cooling water, is required.

Burettes

About half the burettes used in an elementary chemistry laboratory will be damaged in some way in the course of an academic year. The top may be chipped or broken, and, if the damage does not extend to within about 5 cm of the zero mark, a clean straight cut can be made. The new end is melted, slightly thickened and then flared. Otherwise, if the zero mark is intact and will not be affected, a short length of tubing should be joined to the burette top, cut to the required length, thickened and slightly flared.

Burette stopcock keys sometimes freeze into the barrels. They can be removed with a special tool[1] made for that purpose and shown in Fig. 8.1. A small piece of card, placed between the small end of the stopcock key and the threaded steel rod, will help to reduce damage to the key. The above procedure is only

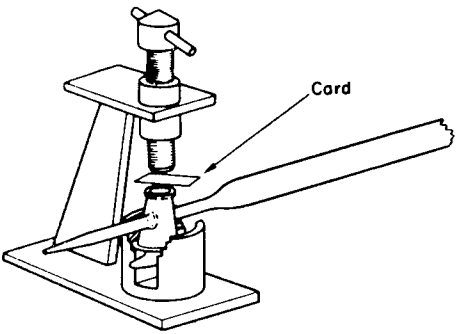

FIG. 8.1. A tool used to remove stopcock keys.

effective if the key has been frozen in the barrel because of lack of grease, or because the barrel has been hotter than the key when they were assembled and has shrunk on to it. Should the stopcock be frozen as the result of chemical attack, by such substances as caustic solutions, in most cases it will be useless to attempt to separate the parts.

Soda-glass burettes are widely used and it is advisable to keep a stock of soda-glass burette stopcocks as replacements. The keys are not, in general, interchangeable. If either key or barrel is damaged a new stopcock must be fitted.

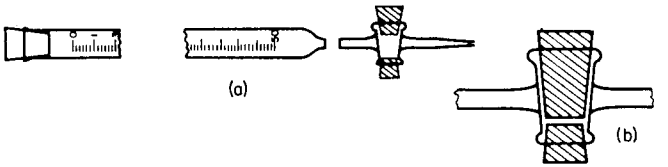

FIG. 8.2. Replacing burette stopcocks. (a) The burette and stopcock ready to be joined. Note the orientation of the stopcock barrel and the scale. (b) Shows the side tube bore blocked by a stopper which is too long.

To join a new stopcock to a burette cut off the damaged one close to the burette tube. This should be done carefully so that a straight, clean end of approximately the same diameter as the stopcock tube is obtained. Now cut a straight end on the wide tube of the stopcock so that not less than 4 cm of the tube is left. Remove the key and plug the barrel with small close-fitting corks. Be careful not to select long corks, as they may block the side tubes and prevent air from passing through. The newly cut tubes are now joined together. Special attention should be given to making a neat join, tapering gradually from one tube to the other. The stopcock barrel should be so orientated with respect to the burette scale that the numbers can be readily seen when the burette is held vertically, with the key handle on the right side, as in Fig. 8.2.

Even with the key removed, stopcocks are somewhat heavier than a similar length of tube. They are also relatively short and

decidedly unbalanced. They are therefore difficult to control and determined efforts will be required before skill and speed is developed in joining them to burettes and other apparatus. Such joins can also be made with the handlamp, using a retort stand to support the burette. This method is slow, so it is better to take time to learn to do the work with the bench burner.

It should be noted that burette and pipette jets are usually of thick-walled glass and that they must be heated up very slowly, and carefully annealed after they have been repaired or drawn out. Further, old soda-glass capillary tube may splinter in the flame no matter how carefully it is heated.

Great care must be exercised at all times to avoid heating the ground-glass barrel of a stopcock. Ground surfaces on soda glass will not survive heating in an air–coal-gas flame.

Measuring cylinders that have been chipped or broken above the graduation marks may be repaired by cutting a straight end on the tube. A straight scratch is made with the knife or file and the end cut off with a hot wire. This end is fire-polished. The spout is formed by heating a suitable area of the cylinder end till the glass is plastic, then gently stroking it into shape. The spout should be located on the left side of the cylinder, when the graduation marks are facing towards the glassblower, with the numbers slightly to the right of the centre line, as in Fig. 8.3.

Should the base of a graduated cylinder be chipped, the sharp

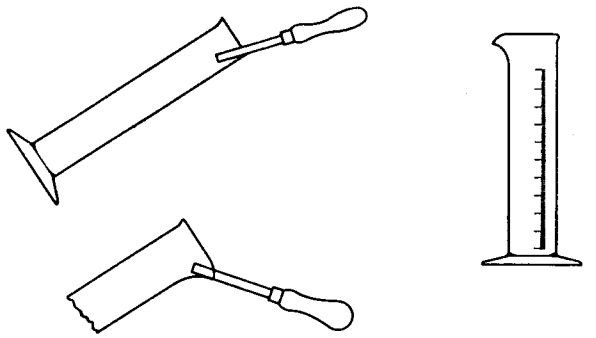

FIG. 8.3. Forming a new spout on a measuring cylinder.

edges are ground off on the side of the carborundum cutting wheel. No other repairs should be attempted, as the base is usually of very thick glass and will not survive heating.

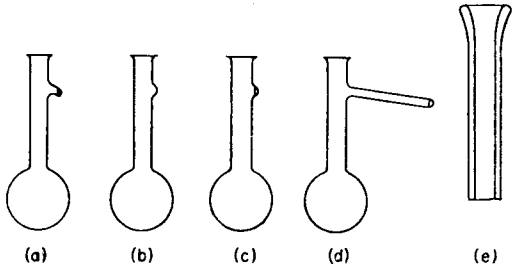

FIG. 8.4. Replacing the side-arm on a distilling flask. (a) Shows the flask and the remnant of the side-arm. (b) The side-arm melted and pulled off with tweezers. (c) A hole blown in the flask neck. (d) The new side-arm joined on. (e) Shows the shape and wall-thickness of the side-arm prepared for joining to the flask.

Buchner flasks are made with a substantial wall-thickness since they must withstand pressure differences of 1 atm. No attempt should be made to repair such flasks unless they can be oven annealed. Flame annealing is inadequate for such heavy-walled glassware; the possibility of failure when evacuated is considerable and the consequences could be serious.

It should be noted that the safety of the user is much more important then any small saving in time or money that may result from repairing damaged glassware. Unless the repair is neat, strong and annealed, then the glassware should not be re-issued for use in the laboratory.

Distilling-flask delivery side arms usually made from tubing of about 6 mm diameter, and are easily broken. These flasks are, or should be, made from borosilicate glass and are easily re-paired. The remnants of the delivery tube should be removed and a neat hole, about 8 mm diameter, blown in the flask neck. A previously prepared tube, 15 cm long, 8 mm diameter and of medium or heavy walled glass, has one end fire-polished and the other end slightly flared, as shown in Fig. 8.4. The flared end is

joined to the flask neck, blown and pulled so that it is inclined downwards, towards the flask bulb. The flask neck, in the region of the delivery tube join, is flame annealed.

Stirrers

There is some demand for stirrers in most chemical laboratories; they vary from simple glass rods with fire-polished ends to centrifugal stirrers and link stirrers, both power driven. Some common types are shown in Fig. 8.5. The flattened end of type (b) is made by melting the end of the rod and gathering the glass into a spherical blob. This blob is then pressed flat with tweezers or between flat carbon sheets. The rod should be pulled very slightly to improve the general shape, but this pulling should not result in any constriction of the glass. Constrictions are always weaker than the rod. The button end, on type (c) is made by pressing a spherical blob of hot glass vertically down on a

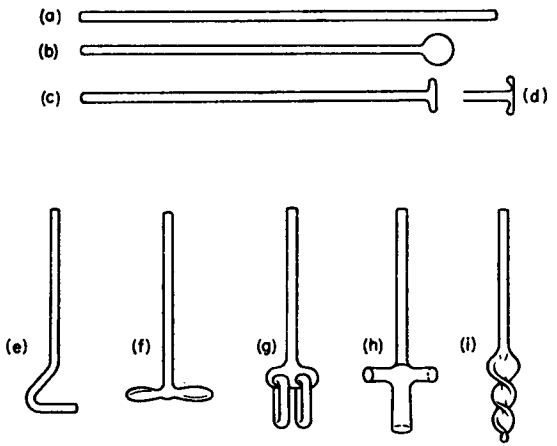

FIG. 8.5. Glass rod stirrers. (a) Plain rod stirrer. (b) Paddle stirrer. (c) Button stirrer. (d) Button stirrer pushed too far down and liable to crack. (e) Bent rod stirrer. (f) Propeller stirrer. (g) Link stirrer. (h) Centrifugal stirrer. (i) Spiral stirrer.

horizontal carbon plate. The button must have a shape similar to that shown in Fig. 8.5c. Should the rod be pressed too far down, so that the shape shown in (d) is obtained, there is some possibility that the button will crack at some later time.

There are many types of mechanically driven stirrers. Bent rods, shown in Fig. 8.5e, are unbalanced and only suitable for low speeds of rotation.

Propeller type stirrers (Fig. 8.5f) can be used at a wide range of speeds and are made in the following manner: the blades are thin discs pressed on the ends of two glass rods and similar to the discs in Fig. 8.5b. The discs are then attached to diametrically opposite sides of a small button formed on the end of the stirrer rod. The joins must be melted thoroughly and the blades adjusted so that they are at about 45° to the centre line of the shaft. The temporary handles are then removed from the blades and the stirrer is flame annealed.

Link stirrers (Fig. 8.5g) are more difficult to make; however, they can be inserted into flask necks. The links swing out when the stirrer is rotated, and effectively agitate the liquid contents of the flasks. Each link is made from a 3 mm rod bent into U-shape. The limbs of the U's are all cut to the same length, usually 35 mm.

A short length of 3 mm rod is joined to the end of the 5 mm stirrer shaft as shown in Fig. 8.6a; the limbs of this 3 mm rod are bent and cut off as shown in (b) and joined to the shaft as shown in (c). A small, pointed flame should be used to avoid distorting the shaft. The holes should be shaped with a pointed carbon tool and should be of such a size that the links will pass through easily (see Fig. 8.6d). The shaft end is then flame annealed and set up to cool.

A link is inserted and held in place with a wooden wedge made from a matchstick. The ends of the link are now softened in the flame and bent towards one another with tweezers. They are then joined and the worked glass shaped into a neat U, with a round carbon rod (Fig. 8.6e).

When the first link is cool it is turned round so that it rests against the shaft and is again wedged in position. The second

link is then fitted and completed in the same way as the first (Fig. 8.6f).

This type of stirrer is best made from borosilicate glass. The shaft end, with the two holes, should be flame-annealed. The links should be carefully shaped or they may stick in the holes and reduce the efficiency of the stirrer.

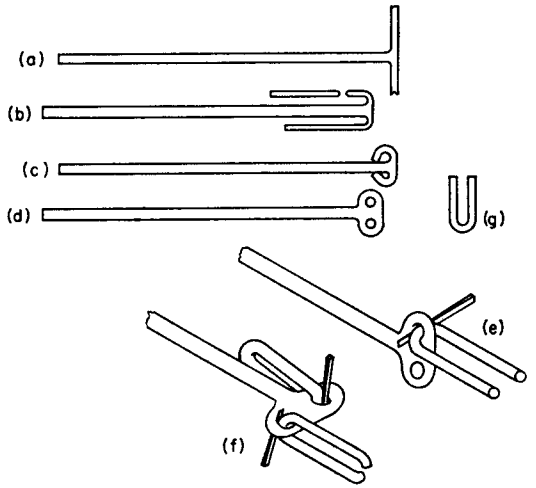

FIG. 8.6. Link stirrers. (a) A T-join made with rod. (b) The ends bent round and melted off. (c) The ends bent further, ready to join to the rod shaft. (d) Shows the completed shaft end. (e) The first link, wedged in place with a matchstick. (f) Shows the first link completed and wedged so that it will be clear of the flame. The second link is wedged in place and its ends bent round ready to be joined. (g) A link of bent rod, ready to be wedged in the shaft end.

Centrifugal stirrers have many uses. When completely immersed, the liquid in the rotating cross arms is centrifuged out and replaced by liquid entering the bottom tube. In making this stirrer the bulb should be strong and neat and the shaft and the bottom, or inlet, tube, must be in line. The cross-arms are cut to exactly the same length, the inlet-tube length is such that the stirrer is immersed in the liquid when rotating, but is not otherwise important (see Fig. 8.5h).

Spiral or worm stirrers are suitable for agitating the contents of test-tubes and boiling tubes. A large sphere of molten glass is pressed into a thin disc by the method described for type (b). A rod is then attached to the disc and the disc heated in a soft flame and stretched and twisted to the spiral form shown in Fig. 8.5i. The first attempt to make such a spiral may not succeed, but repeated efforts will soon produce surprisingly long and regular worm stirrers.

Calcium Chloride Tube

This common item of glassware-stores equipment can be easily made, and provides a valuable glassblowing exercise. Using the method illustrated in Fig. 6.32 (p. 101), join a 15 cm

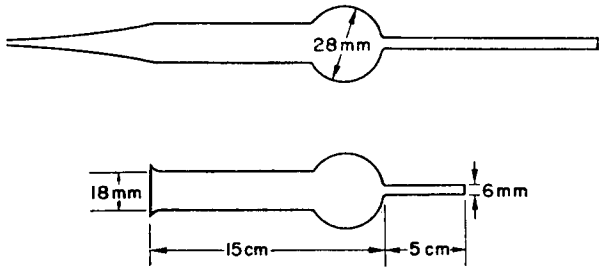

FIG. 8.7. A calcium chloride tube, showing the stages and suitable dimensions for its preparation.

length of 18 mm tube to a 15 cm length of 6 mm tube. Blow a neat, strong, spherical bulb of 28 mm diameter in the larger tube, taking care to avoid melting the smaller tube.The ends are finished as shown in Fig. 8.7.

Thistle Funnel

This type of funnel does not enjoy the popularity it deserves. When used as a filling funnel it will reduce the risk of splashing the liquid poured into it.

They can be made in a wide range of sizes and is one of the best glassblowing exercises, providing an opportunity to practice drawing points, making constrictions, joining tubes of different sizes, blowing bulbs and making flares.

The steps are shown in Fig. 8.8.

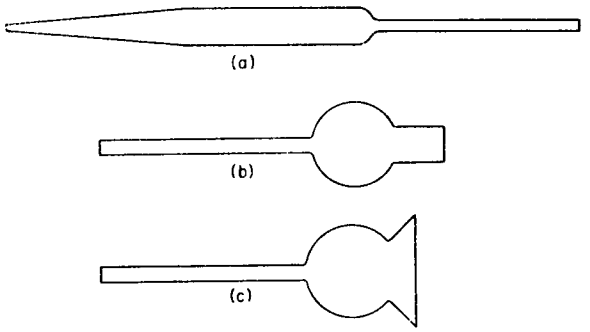

FIG. 8.8. A thistle funnel. (a) Join the stem to a constriction on the larger tube. The point on the larger tube must be straight and of reasonable length. (b) The bulb is blown; note that ample glass must be left to make the flared top. (c) The completed funnel. The flare should be reasonably proportioned and have an angle of about 90°.

The bulb must be strong and neat, the joins should be thoroughly melted, of uniform wall-thickness and free from any constriction. In making the flare be careful not to melt the "waist" or the bulb, as they are easily deformed with the flaring tool.

Hero's Fountain

Hero's fountain is an old and, to some people, puzzling device. The bulb *A* is blown up to about 50 mm diameter and fitted with a jet tube extending almost to the bottom. An 8 mm tube is now joined to the bulb, near the ring seal, and bent over parallel to the jet tube. The jet tube is bent into the shape shown in Fig. 8.9, then constricted and drawn out into a short jet of not more than 1 mm bore. The thistle funnel *B* and bulb *C* are made with 8 mm

connecting tubes as shown in Fig. 8.9. The 25 cm tube is bent into a U with parallel limbs and as near to *C* as possible.

At this stage a check should be made to ensure that the vertical distance from the bottom of funnel *B* to the top of bulb *C* is more than the vertical distance from the jet to the bottom of bulb *A*.

The bends should also be checked to ensure that the horizontal distance from the jet to the 8 mm tube on *A* is the same as the horizontal distance between the parallel 8 mm tubes on *B* and *C*.

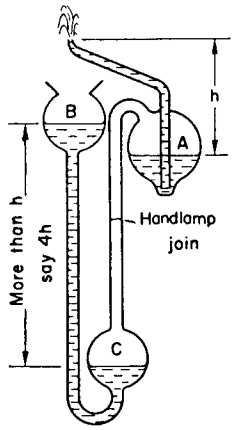

FIG. 8.9. Hero's fountain.

The two parts are now joined together so that the jet is above and in line with the centre of the funnel.

The fountain is now completed.

When bulb *A* and funnel *B* are filled with clean water and *C* contains only air a fountain of water will emerge from the jet and fall into the funnel.

The technique of filling *A* and *B* is left to the reader as an exercise in manipulation.

The explanation of the fountain is a simple matter of hydrostatic pressure and is also left to the reader.

Hero's Engine

This, like Hero's fountain, was first made about 130 B.C. The materials used then certainly did not include borosilicate glass.

A bulb of about 100 ml capacity is blown and tubes of about 5 mm diameter joined to it so that they are exactly in line and symmetrical with the bulb. Two side-arms, 4 cm long and also

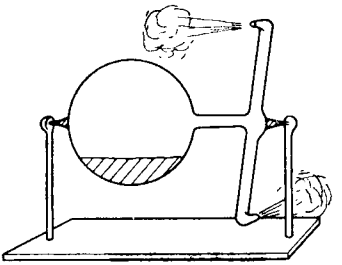

FIG. 8.10. Hero's engine.

5 mm in diameter, each with a good point, are now joined at right angles to one of the bulb spindles so that the side-arms are in line.

The spindles are sealed off, leaving a reasonably thick bearing cone of solid glass. The other members of the two bearings are made by slightly flattening one end of each of two 5 mm rods and pressing a small indentation in the flattened ends with a sharpened carbon tool.

The side-arms are bent at right angles in opposite directions, and the points drawn out to make small jets of equal bore.

The bearing rods may either be joined up to a glass rod stand or inserted in holes drilled in a wooden base (Fig. 8.10).

The bulb should be almost half-filled with water and the bearings lightly greased with vaseline.

Hero's engine will rotate rapidly when the water in the boiler is heated with a soft bunsen flame. If the jet bores are much more than 1 mm the water soon boils away. If, however, they

are very small, then the heating should be done cautiously as a dangerous pressure may build up in the boiler and cause it to explode.

Spinning a Foot

A few small items of glassware are convenient to use, and store, if they are provided with some type of stand or foot. Such stands can be made by bending glass rod into a triangle of convenient size. A more elegant foot, of the type found on wine glasses, can be made in the following manner.

Pull double points on a heavy-walled tube, of about half the diameter of the required foot and with the distance between the shoulders equal to the diameter of the foot. Cut one point open and blow a thick hemisphere at the other shoulder. Now join a 20 cm length of 5 mm rod to the hemispherical end so that the junction is smoothly tapered and free from constrictions and re-entrant angles. The glass near the join is flame annealed and,

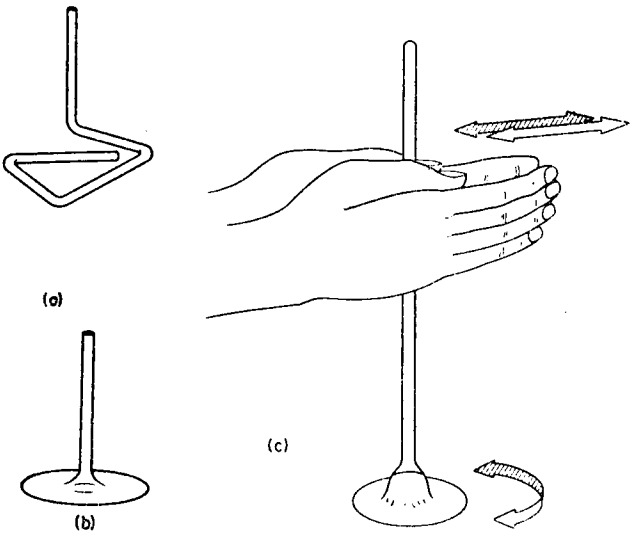

FIG. 8.11. (a) Triangular foot made from glass rod. (b) Wine glass foot made by spinning. (c) Spinning the foot.

when cool, the tube is cut off straight so that the distance from the open end to the shoulder is rather more than the radius of the foot. The open end is now heated in a large hot flame till all the glass between the hemisphere and the end is plastic and the edges are beginning to melt in. The glass is now removed from the flame and the rod held vertically, between the palms of the hands, with the hot glass down. The rod is rolled quickly, back and forth, between the palms. The foot will now spin out into an almost flat disc (Fig. 8.11).

Many of the early attempts at spinning will result in very unsatisfactory shapes. However, determined practice and careful heating and rotation will soon produce satisfactory feet.

Condenser Adaptors

These adaptors are more difficult to make than their simple shape suggests. They should, therefore, be made in batches of 20 or 30 rather than in small numbers.

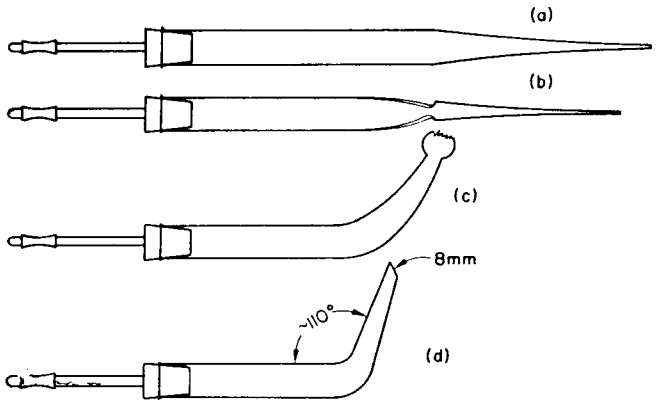

FIG. 8.12. The essential stages in making a condenser adaptor. (a) The prepared tube fitted with an asbestos stopper and plugged tube handle. (b) Shows the glass constricted and gathered. (c) The first stage of bending, pulling and blowing. (d) Finished adaptor. The small end is cut off with a rubber-bonded cutting wheel.

For each pair cut a 35 cm length of 23 mm tube and slightly flare each end so that an asbestos stopper with an 8 mm tube handle will fit neatly and securely. Now pull good points in the centre and cut them open. Each one should now be like Fig. 8.12a.

Mark each tube with the glass-marking pencil or knife at 10 cm from the open end. Now make a constriction, just to the right of the shoulder, of about 8 mm o.d. and with rather greater wall-thickness than the tube. Heat all the glass between the mark and the constriction until it has gathered to the shape shown in Fig. 8.12b.

Rotation is now stopped and the hot glass gently blown and pulled up so that the bend is neat and the worked glass tapers from 23 mm diameter to 8 mm diameter in about 11 cm.

When the adaptor is cool the small end is cut off at about 45° with the cutting wheel and fire-polished (see Fig. 8.12d).

Making Spiral Coils

Helical coils of glass tube make efficient condensers and heat exchangers, since they have a large surface area and the rate of flow of the liquid in the spirals is high and uniform throughout.

They can be made freehand or by winding a hot plastic glass tube on a suitable former.

Condenser coils should be of reasonably accurate helical form and made from thin-walled tubing of 6–8 mm diameter. Successive turns of the coil should be as close together as possible, without actually touching, so that the finished spiral will not be unnecessarily long.

Such spirals are very difficult to make freehand and are normally wound on a former. A straight borosilicate glass tube, covered with asbestos paper, can be used as a former, but some difficulty may be experienced when withdrawing this tube from the finished coil. Brass tube formers are easier to withdraw, provided they are quite straight. Spiral winders with a ground taper finish eliminate these difficulties. They are fitted with a

wooden handle and a small metal loop which holds the glass tube end securely.

Spiral winding calls for skill and concentration. Thin-walled glass quickly overheats and loses its uniform shape, so it is better to practise with medium-walled tube and to make successive turns about 5 mm apart.

One end of the selected tube is bent round into a U, with the tweezers. The short limb of the U should be about 1 cm long, just enough to hook into the metal loop, but not so long that it must be cut off to remove the finished spiral (see Fig. 8.13a).

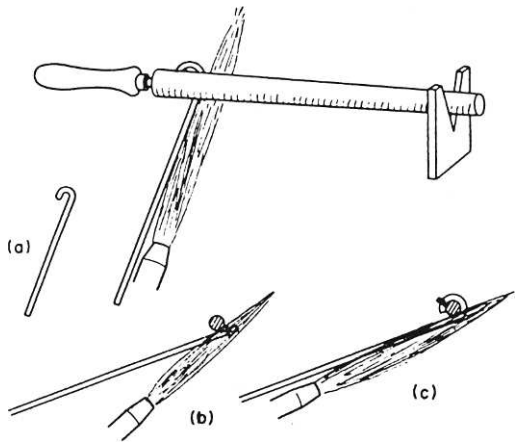

FIG. 8.13. Spiral coils. (a) Shows the small U-bend ready to hook into the metal loop on the forming tool. (b) The elevation of the tube at the beginning. (c) The elevation of the tube when winding is under way.

A long air–gas flame will give just the right temperature to soften borosilicate glass, oxy-coal-gas flames are too hot.

The spiral former is quite heavy and, unless it is supported, will prove very tiring to hold and turn when winding on about 4 ft 6 in. of tube.

The first half turn is the most difficult to make as the glass must be hottest just as it is fed on to the former. The tube must, at this stage, be held horizontally. The former is slowly rotated as

shown, the speed of rotation being adjusted so that the tube winds on just as it becomes sufficiently hot. If rotation is too fast the glass may break. If too slow, the glass will overheat and tend to flatten or stretch. There will always be a slight tendency for the tube to flatten, but if the flame is correctly adjusted, and local heating avoided, then a suitable rate of rotation of the former will result in only slight distortion of the circular cross-section of the glass.

As the tube begins to bend round the former it should be brought down into the flame where it will be gradually heated along the length and will reach the correct state of plasticity just as it bends on to the former.

The glass tube must, of course, be fed on to the former at a slight angle. When skill and experience in judging and maintaining this angle have been acquired, and the flame is so adjusted that it just softens the tube but passes under the former, then winding will proceed smoothly and steadily. (See Fig. 8.13c.)

The next step is to try to wind on successive turns so that they are about 2 mm apart, yet do not touch. Should successive turns touch they will probably stick together. This not only spoils the appearance of the spiral but may cause it to crack, even after annealing.

Finally, thin-walled tubing is wound on. Rather more care will be required and the flame must not be too hot. Winding will proceed somewhat faster and there must be no interruption of the work until only about 15 cm of tube remain in the right hand.

The left hand is used to support and rotate the former. The right hand supports the tube, holding it about half-way between the former and the remote end. This hand controls the angle between the tube and the former, keeps the glass in the flame and guides it on to the forming tool.

When the required length of spiral has been formed it should be allowed to cool for a few seconds. The former is now cautiously rotated in the opposite direction and the spiral held lightly and firmly. This should free the small U-bend from the metal loop and the helical coil can be easily slid off. No annealing is necessary and as soon as the coil is cool enough to handle the ends can be

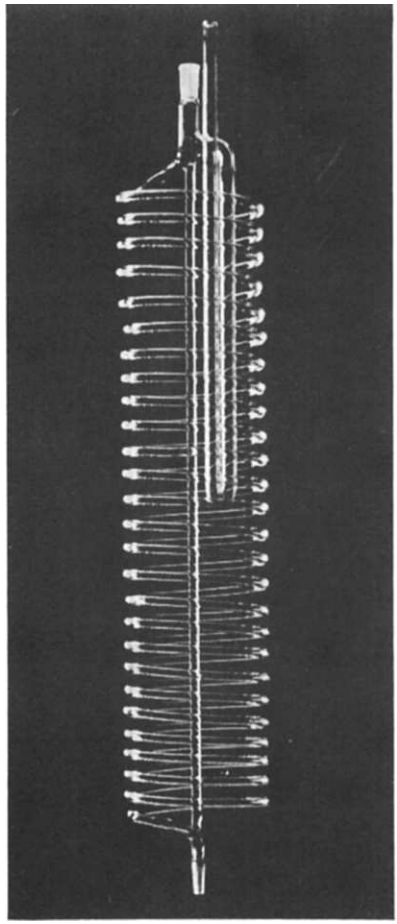

PLATE 8.1. A 610 cm (20 ft) spiral coil for gas chromatography. This coil was made, in five sections, on an asbestos-covered glass tube. The sections were joined together with a handlamp.

prepared for further work in making a condenser or heat exchanger.

There is some limitation to the size of tubing that can be wound on any given former. It has been found that tubing will be appreciably distorted if it is more than one-quarter of the diameter of the spiral former. Since a small amount of distortion, or flattening, of the tube makes no difference to the performance of the spiral and very little difference to its appearance, the ratio of former diameter to tube diameter may be taken as 3 to 1.

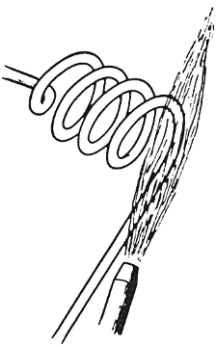

FIG. 8.14. Making a free-hand spiral coil. The glass must be moved and turned in the flame so that the tube is heated all round its circumference.

Free-hand spirals are made without any blowing and without the aid of a forming tool. Some difficulty will be experienced in keeping the internal diameter of the helical coil uniform and in maintaining a constant distance between successive turns.

The first half-turn is made in the tube so that the short limb is about 20 cm long. This limb is now bent round so that it is in line with the centre of the proposed helix. The long limb is heated in an air–coal-gas flame and carefully bent into the required shape. Frequent inspection and adjustment will be necessary to keep the turns uniform in spacing and diameter.

Free-hand spirals are usually much less accurate than those wound on formers, and are therefore seldom used for condensers (Fig. 8.14; Plate 8.1).

They are satisfactory when used in making mercury–toluene thermo-regulators or for any other heat exchanger that does not require an accurate helix.

Should a spiral be required of such dimensions that a 5 ft length of tubing will not suffice, then the join should be made before winding begins. This join must be quite uniform in wall-thickness and diameter, since otherwise it will not bend smoothly and will show up as an obvious defect in the finished coil.

There is plenty of scope for ingenuity in discovering methods of winding helical glass spirals, and nearly all glassblowers have their own methods and equipment. One method involves the use of a glassworking lathe to turn the forming tool. The left hand rotates the chuck, the right hand holds a handlamp and also guides the tube.

Handlamp Work

A handlamp is essential for all jobs that cannot be conveniently rotated in the bench burner flame. For example, it is used to seal off test-tubes containing samples which must be kept cool, or which should not be disturbed. It is indispensable if additions have to be made to glassware which is part of a complicated and semi-permanent apparatus, such as a vacuum line, and for sealing-off evacuated vessels.

A light-weight handlamp, with readily accessible and easily adjusted taps, is best. The orifice-mixing type is most suitable for working soda glass, as it provides a large soft flame for preheating and annealing, together with the fairly wide range of flames so desirable for joining, bending and sealing-off. The rubber tube connection should be 2 m long and of a heavy wall. Thin-walled tubes kink easily cutting off the air or gas and so extinguishing the flame. The connections must be securely made and wired on for added safety.

A 2 m length of 6 mm rubber tube, fitted with a mouthpiece at one end, will be required for blowing up the molten glass. If it is required, a drying-tube containing calcium chloride (granulated) can be inserted in the rubber blow-tube.

PRELIMINARY EXERCISE

Make and check the rubber tube connections to the handlamp from the bench taps for air and gas with all taps closed. Open the bench taps, then turn on the handlamp gas-tap and light the gas. Holding the lamp in the right hand, and using the thumb of that hand to turn the gas-tap, vary the flame through the range from a large bushy flame to a small flame about the size of a candle's. Be careful, at all times, to avoid pointing the flame towards the bench top, clothes or anything liable to be damaged by heat. Adjust the flame to about one-half its maximum size and gently open the air-tap. Practise adjusting both gas- and air-flow so that any type and size of flame can be obtained; that is, any type and size within the limits imposed by the gas pressure and the air-jet diameter.

Whenever possible, handlamp work should be done with the glass tube in a vertical position, since joins and constrictions made in horizontal tube will sag downwards when the glass is plastic and lose their neatness and strength, as in Fig. 8.15.

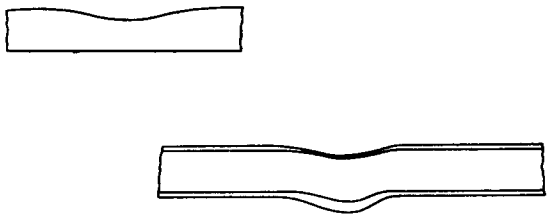

FIG. 8.15. Horizontal tubes tend to sag when worked with a handlamp.

To constrict a tube of about 10 mm diameter, first set it up in a retort stand with about 20 cm projecting above the clamp. Hold the top of the tube in the left hand and heat up the glass about 8 cm above the clamp, gradually increasing the air-flow to give a flame about 15 cm long. The flame should be at right angles to the tube and must be kept moving so that the whole circumference of the tube is uniformly heated. The handlamp should be held lightly but firmly and should be turned in the

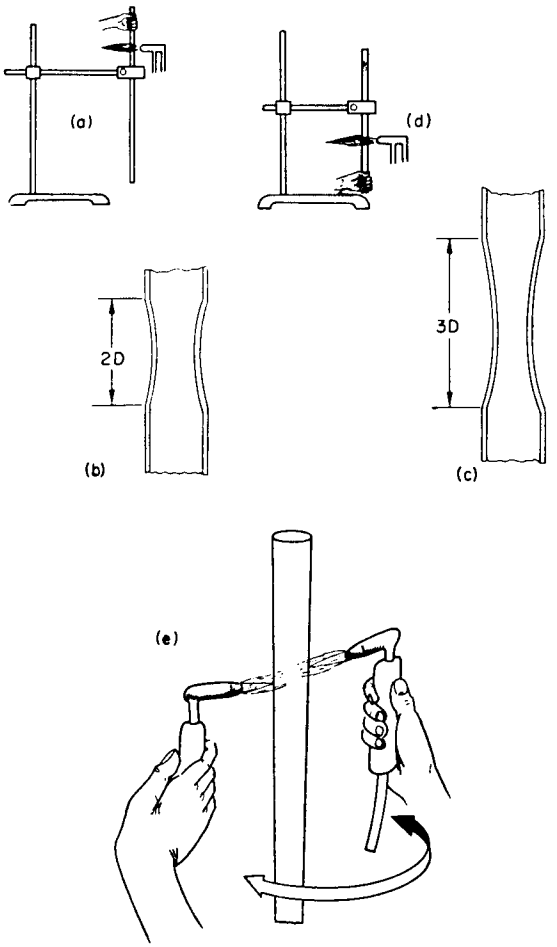

FIG. 8.16. Constrictions made with the handlamp. (a) The tube set up in a retort stand. (b) The glass heated, constricted and ready to pull up. (c) The finished constriction. (d) An alternative arrangement of (a). (e) Shows the movement of the handlamp necessary to heat the glass uniformly all round.

hand so that the flame is always directed on to the glass. The flame moves in a horizontal plane and through about 200° or more. Close attention must be given to heating the glass on the side of the tube remote from the glassblower. Close and continued inspection of the heated glass is essential. The left arm is held well up and out of the line of the flame. The left hand supports the tube above the heated glass, keeping the whole tube in line, and keeping the molten glass free from twists. The glass wall is allowed to thicken and the tube is constricted by carefully raising and lowering the left hand. The handlamp flame must be kept moving round the tube, must be accurately directed towards the glass, and, in the latter stages of constricting, directed towards the lower three-quarters of the molten glass.

The diameter of the bore of the constriction and the wall-thickness of the glass must be carefully observed. The dimensions can only be estimated by inspection (Fig. 8.16).

This exercise should be repeated with the tube inverted in the retort stand. The glass below the proposed constriction is

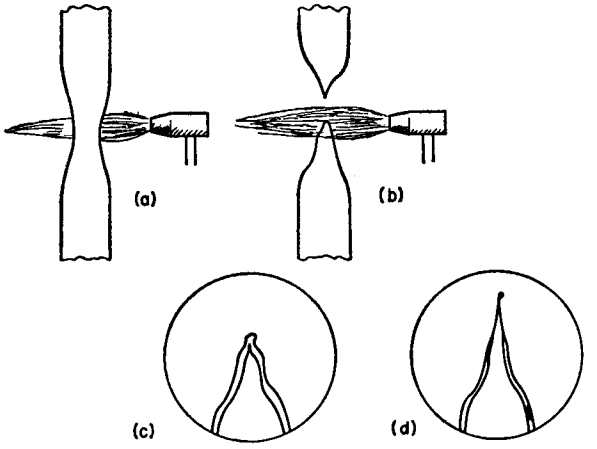

FIG. 8.17. Sealing off a constricted tube. (a) Preliminary heating with soft flame. (b) Sealing off with small, hot flame. (c) A satisfactory seal with no thin glass. (d) The result of pulling the glass up too far. Note the thin fragile glass.

held in the left hand and raised against gravity as the glass softens.

Such constrictions are made in preparation for further work, mainly sealing-off.

To seal-off a constricted tube, the glass is first heated up with a fairly large soft flame till the constriction is just plastic. The handlamp is now adjusted to give a small, sharp-pointed flame and the glass just above the middle of the constriction heated till it constricts further and finally closes. Very little glass should be melted, and the wall-thickness should be kept up to that of the original tube. Do not be tempted to pull up the tube above the constriction, allow the flame to do the sealing. Although thin-walled seals will withstand fairly high internal pressures, they are very easily broken (Fig. 8.17).

Handlamp Joins

VERTICAL JOINS BETWEEN TUBES OF SIMILAR SIZE

The largest part of the glassware is supported in a retort stand so that the tube to be joined is vertical and at eye-level whether the glassblower sits or stands. Both tube ends should be clean and cut straight. The shorter tube is held in the left hand so that it is in line with the other tube and about 5 mm from it. (See Fig. 8.18.)

Both tube ends are now given a preliminary heat up with a soft flame and then melted, lightly touched together and pulled very slightly apart.

The handlamp must be kept moving throughout the whole operation so that the glass is uniformly heated.

Heating, with a small moderately hot flame, is continued till the join is fused together, and the glass has gathered up to the original wall-thickness again. Meanwhile the glass tube diameter is recovered by repeated gentle blowing with the mouth. The glass below the join is then flame-annealed.

Care must be exercised to avoid holes or gaps in the join. The

hot glass should be blown to maintain the tube diameter but it should not be blown up more than this as pulling to reduce the diameter may result in thin-walled glass.

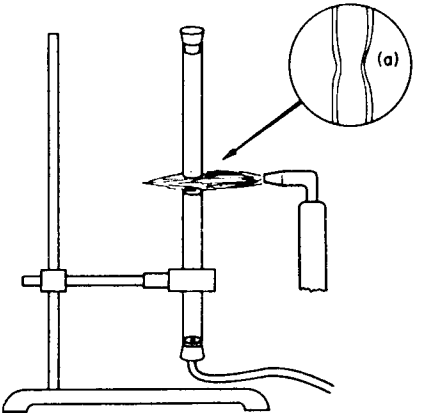

FIG. 8.18. Handlamp join showing the retort stand and blow tube. The stoppered tube is held in the left hand; the handlamp is held in the right hand and moved round to heat all the join. (a) shows the join pulled slightly apart immediately after the ends are touched together.

After some practice in using the handlamp, selecting the flames, and controlling the glass with the left hand, it will be possible to so time the joining that it will be completed when the glass wall-thickness and tube diameter have just been recovered.

If both ends to be joined are attached to large or otherwise unwieldy glassware, then both sections are clamped so that the straight, clean ends are almost touching. A rod, of the same type of glass as the apparatus being made, is drawn out into a long, thin point about 2 mm in diameter.

The tube ends are heated all round till a sodium yellow colour is seen on the glass surface. The handlamp is quickly adjusted to a small, hot, needle flame. The end of the thin rod is melted into the space between the tube ends and progressively fed into the whole circumference of the join (Fig. 8.19).

The point of the flame should be directed mainly on the rod, which will then be somewhat hotter than the tube ends. Should the tube ends be overheated, then they may melt sufficiently to run back and an enlargement of the space between them will appear and be difficult to close up.

It is, of course, essential that the rod be fused into the tube ends, but the first step is to close up all the space round the join so that air pressure from the mouth can be used to maintain or recover the circular cross-section of the tube.

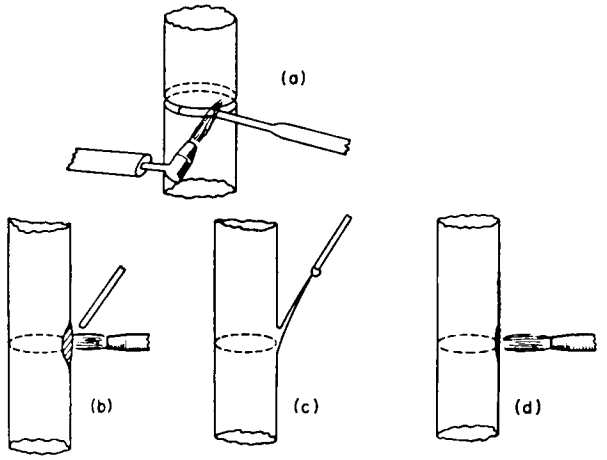

FIG. 8.19. Handlamp join using rod fill. (a) The rod being fed into the space between the previously heated tube ends. (b) and (c) Removing a thick spot from join. (d) Remelting the spot to recover the shape.

Some care must be exercised, throughout the application of the rod, to avoid any excess thickness of melted glass. Should thick spots appear they must be locally heated and pulled upwards with a rod or tweezers. The resulting thin point is then melted off, near the tube wall, remelted and blown till the shape and wall-thickness of the tube is recovered.

Every effort must be made to confine the heating, and therefore the melting, to the minimum length of tube. No vertical

movement of the upper tube is possible and excessive heating will cause the glass to run down under gravity, leaving a thickened ring of glass under one that is thin and therefore mechanically weak.

The best joins are those that are made without delay. Should any interruption occur in feeding the molten rod into the space between the ends, the whole join must be reheated to avoid cooling cracks.

T-joins can be made with the handlamp, and this is a very convenient way of adding side tubes to large or otherwise unwieldy glassware.

The glassware is set up in a retort stand so that the clamp is at a convenient distance from the proposed working region. The tube to which the side-arm is to be attached should be, wherever possible, horizontal. A rubber blow-tube is connected to the glassware and all other openings are securely stoppered; a trial blow into the mouthpiece, to find out whether there are any significant leaks, is always worth while.

The side-arm is prepared by slightly thickening and flaring the open end. It should either have a strong point on the other end or be about 20 cm long, so that the left hand, which will support the side-arm, can be kept clear of the flame and away from the hot glass.

The next step is to blow a hole in the tube. This hole should be about the same diameter as the side-arm and in the centre of the tube; some care will be necessary to make it neat, in the right place, and of the desired size.

The prepared side-arm is held vertically over the hole and about 5 mm above it. The flared opening and the edge of the hole are heated simultaneously with the handlamp flame; the flame must be kept moving through about 200° and the side-arm should be rotated, back and forth, so that the edges are uniformly heated and softened. When both edges are seen to be uniformly melted and have constricted to about two-thirds of their original diameter they are pressed lightly together and the side-arm immediately pulled up a short distance. Heating is continued until the join is smooth and the glass has gathered slightly.

The molten glass is now blown and pulled up so that it is free from constrictions and thick regions. (See Fig. 8.20.)

The T-join should now be flame-annealed; the vertical tube has been heated all round its circumference and does not require much attention; the horizontal tube has been melted on the top only and it is essential that it be heated all round and that this heating extend on each side of the T-join. Handlamp joins are usually made with a small, hot flame so that the heating is of a local nature, and annealing must always be carefully done.

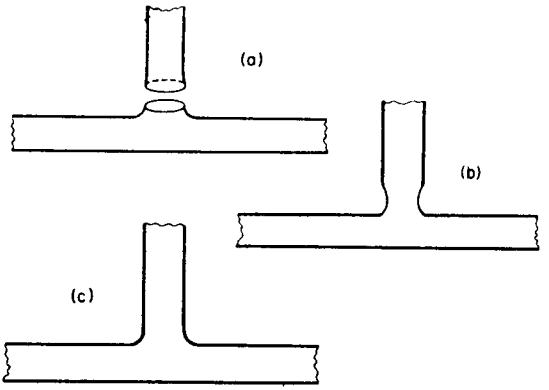

FIG. 8.20. T-join made with the handlamp. (a) Shows the side-arm and the cross-piece ready to be joined. (b) The join pulled up immediately after it is first made. (c) The finished join; the glass has been freed from irregularities in wall-thickness and diameter.

There is always a possibility that the join will take some time to complete and that prolonged working will soften more of the horizontal tube than is desired. The tube may then sag under its own weight. This trouble will disappear with accumulated experience and skill in preparing the members of the join and in using the right flame. The join will then be completed very quickly.

Handlamp work is easier than bench-burner work, since the glass remains more or less stationary. None the less, much practice is necessary before a high standard of work can be turned

out. Many glassblowers begin to learn this work by making joins with the handlamp, using tubing about 5 mm in diameter. The author has made elaborate double outline letters in 15 mm tubing, for neon signs, complete with electrodes and pump stem, using a handlamp only.

When one becomes expert in the use of the handlamp there is a tendency to do many jobs with it which could be done more neatly and in less time if the bench burner were used. Handlamp work involves setting up the glassware in a retort stand, and is usually slower than bench-burner work.

Side tubes can be joined to vertical tubes in much the same manner; the join must be completed quickly to avoid overworking, as this will result in the molten glass sagging down under gravity. Practice is necessary with this type of join, as it is with all glassblowing operations.

Large diameter T-joins in borosilicate glass can be made with the handlamp. Since it is very difficult to heat large diameter tubes uniformly, the hole in the cross-limbs should be blown to the required size and shaped to a neat circle with a carbon rod. The short limb should be clamped so that the open end and the prepared hole are almost touching. The space can be closed up with small diameter rod in the manner described on pages 156 and 157. Large diameter T-joins require very careful annealing.

Liebig Condenser

Preparation

The six component parts of a Liebig condenser should all be prepared before the assembly of the parts is undertaken (Fig. 8.21a). The condenser should be made to standard dimensions[2], unless a non-standard condenser has been requested.

The condenser jacket has two spherical bulbs. These bulbs should have the same wall-thickness as the jacket tube. If they are too thin they will distort when the ring seals are made and when the water tubes are attached. The distance between the

bulbs should be such that the inner tube ends will seal to them without any noticeable alteration to their spherical shape.

The inner tube should be cut to the specified length, or to a convenient length to suit the jacket bulbs if they are other than the required distance apart; the inner tube length is the effective length of the condenser. One end of the inner tube is fire-polished only, the other end is slightly flared.

The water tubes and the vapour inlet and outlet tubes are prepared in the usual way. The inner tube is held in place with corrugated paper. This paper must be of such a length and thickness that it can be withdrawn easily when the first end of the condenser has been completed. It must, however, hold the inner tube in position, concentric with the jacket and with the flared end just touching the jacket bulb. The inner tube may tend to take up an eccentric position because the edges of the corrugated paper are easily crushed. This tendency can be overcome by cutting the paper in half lengthways. The cut edges will then be diametrically opposite and will neutralize any tendency to eccentricity.

Fabrication

When the first ring seal has been made, the glass within the inner tube end is blown out to a neat hemisphere of normal wall-thickness. The water outlet tube is then joined to the bulb and bent over and to one side of the jacket so that the point handle is at 45° to the condenser centre line. This bend must be neat and strong, and as close to the bulb as possible, since water tubes are subjected to quite large forces when rubber tube is attached and detached from them.

The hemisphere is now blown out and the vapour inlet tube joined on. The join should taper smoothly from the inner tube to the vapour inlet. The condenser end is now annealed and set up to cool.

After removing the corrugated paper cylinder the condenser is fitted with a cork stopper carrying a glass tube handle with a side-arm. This side-arm is connected to the water outlet by a con-

venient length of rubber tube, such an arrangement equalizes the pressure on both inside and outside the inner tube and greatly assists in maintaining its shape and wall-thickness (Fig. 8.21).

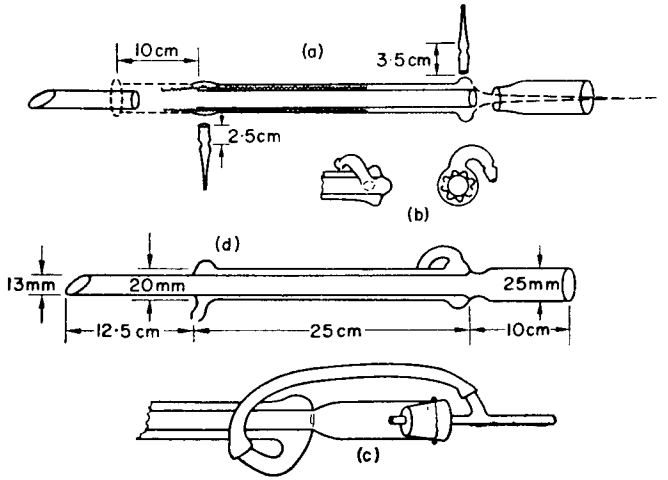

FIG. 8.21. A Liebig condenser. (a) Shows the component parts and the corrugated paper sleeve supporting the inner tube in the jacket. (b) Details of the shape of the water outlet tube are shown in both elevations. The corrugated paper sleeve, in two parts, is shown in section. (c) A blowing arrangement used to make the second internal seal. (d) The finished Liebig condenser.

The extension beyond the second bulb is now drawn off and the second ring seal made. The water inlet tube is now attached so that it is parallel to and on the opposite side from the outlet tube.

Finally, the condenser drainage tube is joined on and annealing should follow the procedure described in Chapter 6, p. 120. The drainage tube is cut off at about 45° to the centre line and fire-polished.

Efficiency

The thermal efficiency of simple glassblowing operations has been measured by Long.[3] He found that, in the simple operation of pulling points, the heat usefully employed was about

1% of the heat generated by the fuel gas burned. Borosilicate glass was used, and preliminary heating would be either brief or entirely absent; annealing would be unnecessary.

While this aspect of a laboratory glassblower's work is not an important one, none the less it is clear that every effort should be made to economize on fuel gas. The work should be carried out quickly and neatly and all reworking should be reduced to a minimum. When annealing time is included and some reworking is required to improve shapes or correct errors, it is conceivable that the thermal efficiency of such jobs as making a Liebig condenser could be as low as 0·1%.

References

1. A. Gallenkamp & Co., London, 1964 Catalogue.
2. BSS 1848 (1952).
3. LONG, V. D., Some aspects of the fuel technology of glassworking, *Glass Technology*, 1965, pp. 124–35.

CHAPTER 9

Advanced Glassblowing

ALL glassblowing, no matter how simple or complex, consists of various combinations of the basic operations described in Chapters 6, 7 and 8. However, it is desirable that experience be acquired in making glassware of gradually increasing complexity before undertaking to make apparatus with numerous internal seals and incorporating expensive stopcocks and interchangeable ground-glass joints of large size. Some other accessories, such as graded seals and glass-to-metal tubular seals, may not be replaceable at short notice; consequently they should be built into apparatus by experienced glassblowers, who will be unlikely to damage them beyond recovery.

The sustained effort required to perform a variety of basic operations, one after the other, all to a high standard of perfection and in a reasonably short time, is beyond the energy reserves of all but glassblowers with two or three years' experience.

An accurate scale drawing of the finished article is almost essential. This drawing should show all the dimensions and tolerances. The glassblower should beware of requests for "minimum" dimensions, as they rarely include reasonable distances between ground-glass members, and sometimes overlook the dimensions of the joints and stopcocks themselves. He should also discuss with the student or scientist any vague dimension, such as "between 25 mm and 40 mm" and draw attention to the very different capacity/unit length of the extremes of this kind of tolerance.

The preliminary planning of the sequence of operations, and the methods to be employed in supporting internal parts of glassware, should be given careful thought.

Blowing arrangements must be decided in advance. There is little time for improvisation when the work is well under way, when the heated glass is ready to be blown up and no convenient provision has been made for blowing.

If a number of attempts to make a piece of complicated glassware end in the waste bin, the glassblower should consider whether his preliminary plan is adequate.

The finished article should always look like a new article and not like a repair job.

The requests for special glassware brought to the workshop depend entirely on the nature of the work being done in the departments served by the glassblower. These requests are quite unpredictable.

One cannot acquire skill and experience in any particular type of work unless there is a regular demand for apparatus involving that work. The author would be happy to make tubular glass-to-metal seals with the new alloys and matching glasses, but cannot do so until there is a need for them. Nor can he obtain the necessary materials, since there is no justification for them.

Glassworking Lathes

These are machine tools designed to rotate glass tubes so that they can be uniformly heated to such a temperature that joins can be made, bulbs blown and internal seals fabricated. To effectively carry out such operations a glassworking lathe must be of precise and robust construction. It must be fitted with two chucks, rotating at exactly the same speed about a common centre line, and at an easily variable distance apart.

A reasonably high rotational speed is required for flanging and shaping open tube ends, and for blowing bulbs. At such speeds hot plastic glass of large diameter, say 100 mm, will tend to centrifuge out, and this will add to the difficulty of making smooth joins and constrictions. The lathe should therefore have provision for speed control. This can be a continuously variable control, between an upper and lower limit, or a simpler and cheaper three-speed adjustment.

Such a machine is obviously a precision engineering product and will therefore be costly. The purchase of a glassworking lathe should be postponed until the high capital expenditure is justified by the need for reasonable quantities of glassware of large dimensions. Small bench-mounted lathes[1] have a limited capacity and it is doubtful if the time spent in fitting the glass in the chucks, and later removing the finished glassware, would not be better spent in acquiring speed and skill in handwork. They are, however, useful if glasswork must be done by semi-skilled operators.

Where there is a justifiable need for a glassworking lathe, one of large capacity, both in diameter and length, should be considered. Such a machine, with the right accessories, will handle a wide range of tubing sizes. This versatility will outweigh the greater initial cost.

A glassworking lathe, or any other machine tool, has a very large production potential, consequently it has a greater value than any of its products, and should never be subjected to rough treatment. The location of the glass tubes and the flame, or flames, should always be such that the chucks, and other parts of the lathe, are never exposed to excessive heat. At the risk of labouring the point, it should be noted that any glassworking operation whose performance will endanger the lathe, or any of its accessories, must be replanned to eliminate the hazards, or abandoned altogether.

While it is true that access to a glassworking lathe will considerably reduce the hard work involved in making glass apparatus from tubing of relatively large dimensions, it must be borne in mind that new skills must replace hand rotation and manipulation of the tubes. It is essential that all the component parts of any piece of glassware, to be made in the lathe, should be carefully prepared. The dimensions must be reasonably accurate, cut tube ends should be quite straight and clean, flanges should be neat and of the desired wall-thickness, and constrictions should be of the required shape and diameter. The assembly, in the lathe, of complex glassware, requires close attention to joining, sealing and blowing, all of which

are less difficult if the separate parts are accurately prepared.

The work will proceed smoothly and efficiently if due attention is paid to planning the sequence of operations.

Burners

The burners should include crossfires as shown in Plate 4.4. Crossfires are excellent for joining large diameter tubes since the heat is applied all round the circumference of the join, and the length of tube heated is no more than that required to effectively melt the glass.

A bench burner, adapted for attachment to the burner carriage, can be used to work tubing which is too small in diameter for the crossfires, or to heat a greater length of tube than can be conveniently heated with them.

A handlamp, with a selection of three interchangeable jet sizes, will be required for local heating; it is indispensable for joining on side tubes or closing up small holes. Flame annealing must be done in the lathe and a handlamp is often the best tool for this purpose.

Other accessories

The manufacturers'[2] specifications and lists of accessories must be studied, and wherever possible the types of lathe accessories envisaged should be seen in operation before any decision to purchase them is made. Lathe accessories are expensive.

Chucks are available in a number of different patterns. Selection of the most suitable types can be difficult to make, as the manufacturers do not list the advantages and disadvantages of the various types. Scroll-type chucks, with steel or hard asbestos-sheet jaws, will hold straight tubing firmly and centrally. They are liable to crack slightly bent tubes and tube ends, if the latter are held within the length of the jaws. They are unsuitable for holding bulbs or flasks.

Sun and planet chucks have tubular jaws, covered with resilient sleeves of woven asbestos cloth. They are most convenient for holding bulbs and flasks, and rarely damage tubes. However, long lengths of tube, supported in sun and planet chucks, tend to rotate off-centre. Because there is considerable backlash in the gears used to move the chuck arms, they can be difficult to adjust so that all tube sizes are held concentrically.

Vacuum chucks can be used to hold relatively large spherical flasks. It is doubtful if they would be necessary in a laboratory glassblower's workshop, since very little repetition work involving large flasks is likely to be done there.

A tool-bar is a worthwhile accessory. It is fitted to the back of the lathe and can be used to mount a carbon paddle, or a diamond-tipped glass-tube cutter. The tools are manipulated by a lever attached to the tool bar.

Basic Operations

Cutting clean ends

Large diameter tubes, that is, from 30 mm to 100 mm, can be cut with a rubber-bonded carborundum disc as already described. The cut edges, though perfectly straight and free from irregularities, will show up in joins as a grey line. This grey line can be reduced if the cut edge is washed in running water and thoroughly melted before the join is made; but it cannot be completely eliminated.

Another method for cutting straight ends, which leaves almost no mark when the ends are joined, will now be described.

The tube is mounted in the lathe and a straight circumferential scratch made on the outside surface by holding the edge of a tungsten–carbide knife against the rotating tube. Alternatively, the scratch may be made with a suitably mounted diamond point.

A small, hot, pointed flame from the handlamp is now directed precisely on the rotating scratch and heating continued till the

flame begins to show a yellowish colour. The flame is immediately removed and a wet cloth applied to the heated scratch line. The tube will crack cleanly round the line. Some practice will be required to make a straight mark. The author uses a diamond point mounted on a rod attached to the tool bar, as shown in Fig 4.8 (p. 36). The heat must be applied to cool glass and must be confined, as far as possible, to the scratch. Too early an application of the wet cloth will not initiate a crack and overheating may result in random cracks running in all directions.

This method of cutting glass can be used for all types of tubing except silica, which must be cut with the carborundum disc. High-expansion glasses, such as soda glass, will crack spontaneously and timing is unimportant. In most cases borosilicate glass does not crack until the wet cloth is applied and so it is important that the heating be of suitable duration. For example, to cut a 100 mm diameter Pyrex tube with a 3 mm thick wall and rotating at 50 rev/min it is necessary to continue heating for about 20 sec. A shorter time is required for smaller tubes and also for thinner walled tubes; a 25 mm tube with a 1·5 mm wall will crack after about 8 sec. The tube will be easily adjusted without moving the diamond if it is mounted in the tailstock chuck. A wooden rod of suitable diameter mounted in the headstock chuck and passing through the tube will prevent the short lengths, when they are cut off, from falling on the burner carriage, or on the floor.

Pulling points

A lathe with a sufficiently long bed can be used to pull good points in large diameter tubing. The tailstock must be capable of moving away from the headstock over a distance of not less than 0·5 m, otherwise the chucks may become heated (and this should never be allowed to happen); or the points will be of inadequate length.

The tube is mounted in the lathe so that the distance between the jaws of the two chucks is about 30 cm. The chucks must take a firm hold of the glass, but must not be tightened up so

much that the tube is crushed. If the tube is not sufficiently straight to allow the jaws to take a firm, even hold of the tube, then it may be wrapped with a whole number of turns of asbestos paper before being placed in the chucks. The location of the required shoulder should be marked on the tube. The flame must be directed on to the glass so that the mark appears on the shoulder after the point has been pulled.

The choice of flame or flames depends on the available burners. The aim should be to heat a length of the tube equal to about twice the tube diameter. A large flame, from a suitably mounted bench burner, is most satisfactory. Crossfires will also serve provided that the flames are so arranged that the heat is uniformly spread over the required length of tube.

When the heated glass is sufficiently plastic the flames are extinguished and the tailstock moved away from the headstock. The rate of movement is important and some experience is necessary. Too fast a pull will result in points that may be unnecessarily long and thin-walled. The tail stock may reach the limit of its travel while the hot glass is still plastic, the glass may then sag, and the points will be eccentric and unsuitable for handwork. Should the pull be too slow, then the glass may cool and set before the points are of the desired length. As in pulling points by hand, the rate of pulling should be so controlled that the shoulders are at the required distance apart just as the glass sets. A slight tension must be maintained on the drawn points until the glass has lost all plasticity.

Constrictions can also be made in tubes with the aid of the lathe. A carbon rod of suitable diameter will speed up the constricting if it is pressed gently against the plastic glass. Some care must be taken to ensure that the narrowest part of the constriction does not have a greater wall thickness than that of the original tube. Should this occur, there will be some difficulty in making a straight cut at the constriction. It is also necessary to melt a length of glass not less than the tube diameter, otherwise the constriction may have thin walls and some extra work will be required to recover the original wall-thickness.

Flanging

Provided that tube-ends are cut quite straight, and that the wall-thickness of the glass is uniform all round, neat and accurate flanges can be formed in the lathe with the aid of a carbon rod. A bench burner will heat tubes up to about 40 mm diameter. When the glass is sufficiently plastic the flame is removed and the carbon tool used to flare out the tube end. The tool is held in the hands and can be supported on some part of the burner carriage. Crossfires should be used to melt large tubes and some care taken to ensure that sufficient glass is heated to make the required flange. Some practice will be necessary before flanges can be made with a single heating and application of the tool. Very little extra glass need be gathered to make a flange intended for a ring seal or internal seal, but if a flat flanged joint blank is contemplated, sufficient glass must be gathered before the flanging is undertaken. The flange must be strong and allowance made for grinding. A finished thickness of more than 2 mm is desirable.

Glassblowers develop their own flanging techniques, the only criterion being the finished article and no effective method can be considered unorthodox. The author uses a long carbon rod and rests one end on the movable burner carriage when forming a flat annular surface.

Bulbs

When blowing bulbs the wall-thickness of the finished work will depend on having the correct amount of glass melted. Until some experience has been acquired this amount should be calculated and marked off on the tube in the manner described for mouth blown bulbs, in Chapter 5.

Wherever possible, short points or drawn out constrictions should be used to make the rubber tube connection between the glass and the blowing swivel. Should it be necessary to blow through an open end, the latter must be stoppered with a cork fitted with a short length of glass tube. When the glass has been heated, and gathered by moving the tail stock, then the flames

are extinguished and the bulb blown to size. The shape can be modified by further movement of the tail stock.

When large-diameter tube is worked in the lathe it is held, of necessity, in a horizontal position. The air inside is heated by convection and soon becomes hot enough to melt rubber stoppers and to char cork stoppers. Heat radiated from hot constrictions or test-tube ends has the same effect. Rubber stoppers should be avoided in lathe work. If the tube is of large diameter or short length, and a stopper must be used, then the inside surface of the cork should be protected from the heat by an asbestos or metal foil disc. Asbestos stoppers would be ideal, but they are not available in sizes much above 50 mm.

Every effort should be made to ensure that there are no air leaks anywhere in the blow-tube, swivel or stoppers. The stoppers must be a good fit and unlikely to blow out. It is most annoying, when the glass is heated and ready to be blown up, to find that there is a leak, or that a stopper has fallen out.

Straight joins

The procedure used to join tubes of the same size, in the lathe, is essentially the same as that used to join tubes by hand. The lathe does the turning, hand-controlled movement of the tail stock chuck adjusts the wall-thickness, and mouth-blowing recovers the tube diameter. Crossfires are better than a single-flame burner. The former are recommended for tubing of 40 mm diameter, or more; the latter is adequate for smaller sizes. It should be noted that 100 mm tubes can be joined together in a straight line in about 5 min provided that the flames are sufficient in number and of the right size and that the oxidant-fuel gas mixture is suitably adjusted. Inadequate flames cannot melt the glass effectively and prolonged heating with such flames will not produce a smooth, neat join.

In lathe-work, as in all glasswork, the aim should be to complete the operation to the highest possible standard in the minimum time. This approach to glassworking will keep the cost low

and, rather more important, will keep the quality high. With this aim in mind every effort should be made to increase efficiency; to avoid any action which is unnecessary, or which will have to be reversed afterwards, or which makes no real contribution to the finished article.

For example: nothing useful is achieved, when making a straight join in the lathe, by melting more glass than is necessary. Nothing is achieved if the glass is gathered up to a greater wall-thickness than that of the tubing. Nothing is achieved by pulling the melted glass so that the wall-thickness is much less than that of the tubing wall. Lastly, much time will be lost if the join is blown up, and must then be melted back to the original diameter.

To join tubes of roughly the same diameter the smaller one can be flared out to the same size as the larger one before making the join. Should the difference in diameter be considerable, and the smaller one be not less than about 15 mm in diameter, an accurate constriction in the large tube will be the best procedure. If the small tube is less than 15 mm in diameter much time will be saved by making a hemispherical end on the large tube and blowing a suitable hole in the centre of that end; the join can then be made with a single flame burner or a handlamp.

Ring-seals and internal seals can be made with surprising ease, provided the parts are carefully and accurately prepared and the internal parts are adequately supported.

Corrugated-paper sleeves, consisting of a whole number of turns of corrugated paper of sufficient thickness to fill the annular space between the inner part and the jacket, are frequently used. Their employment is limited to reasonably long apparatus since the paper will char and emit smoke if it is too near the heated zone. The sleeve length should be such that the end projects beyond the inner part and so can be used to withdraw the paper when the ring seal is completed.

Asbestos tape or paper is used to support short internal parts. The asbestos must first be heated in the burner flame, since new asbestos tape and paper emit smoke when they are first heated. Again, provision must be made for withdrawing the packing when the glassworking is finished.

Making Dewar flasks

The glassblowing operations employed in making Dewar flasks are not very difficult. However, since these flasks are often used as containers for liquid air, are evacuated, and are sometimes of large capacity, a detailed description of their fabrication is justified (Plate 9.1).

Borosilicate glass is recommended as it is able to withstand the wide range of temperatures involved in using liquid nitrogen. The author makes such flasks from English Pyrex brand glass tubing, and employs a glassworking lathe.

Assume that the nominal capacity of the Dewar flask has to be 1500 ml, and that the largest size of tubing available is 100 mm in diameter; an 80 mm tube will allow a reasonable annular space of about 7 mm. Allowing for a wall-thickness of 2·5 mm in this tube, its internal diameter will be 75 mm and the length of this tube, with a hemispherical end required, can be calculated from the relationship

$$\text{Capacity} = \pi r^2 l + \frac{4}{6}\pi r^3,$$

where r is the internal radius of the tube and l is the distance from the ring seal to the shoulder of the hemispherical end, all dimensions being in centimetres.

Substituting 1500 ml for the capacity we have $l = 31{\cdot}5$ cm.

The first step is to make a test-tube from 80 mm tube, 31·5 cm + 4·5 cm long; the 4·5 cm including 1 cm for the ring-seal flange and 3·5 cm for extra capacity.

The round bottom of this 36 cm test-tube must be neat and strong so that it will withstand a pressure difference of 1 atm.

The outer jacket is a similar tube, 100 mm in diameter and 35·0 cm from the shoulder to the open end. Both tube ends should be cut straight. About 15 cm of 15 mm tube are joined to the bottom of the jacket, this tube being the pump stem. The constriction necessary for sealing-off the evacuated flask is made at this stage; it must be neat and strong, with about 2 mm wall-thickness and 2–3 mm bore.

The inner tube is supported, by two coils of asbestos tape, on a straight steel rod or tube, about 2·5 cm in diameter and about 40 cm long. This steel tube is mounted in the tail stock chuck and the asbestos tape adjusted so that the glass turns true and is firmly held in place.

The outer jacket is mounted in the head stock chuck and a rubber tube and swivel connected to the pump stem.

The tail stock is moved up so that about 1 cm of the inner tube projects from the jacket, as in Fig. 9.1.

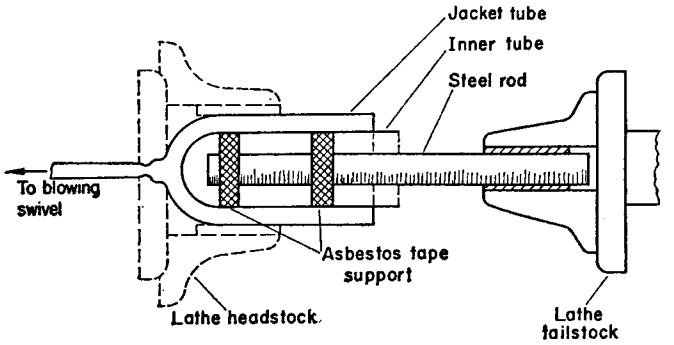

FIG. 9.1. Dewar flask mounted in lathe chucks with the inner tube projecting from the jacket.

The crossfires are directed on to the open tube ends and when the projecting end is workable it is tooled out until the flange touches, and is gently pressed against, the jacket end.

The crossfires are moved back, and the join melted together and blown into shape. Excessive working should be avoided by using adequate heating and by blowing just enough to form a neat join of uniform wall-thickness. The flask should be oven-annealed.

SILVERING

It is essential that a Dewar flask be silvered if it is to be used as a liquid nitrogen (LN) container. Unsilvered flasks have the

advantage of transparency, but absorb heat from the surroundings and the contained refrigerant soon evaporates.

The method described by Wheeler[3] has been found to be satisfactory. Attention is drawn to the importance of having clean glass surfaces. It is recommended that the surfaces be thoroughly cleaned before the assembly of the Dewar, and that, when clean, those surfaces be untouched by hand. Fingerprints can prove troublesome.

The method of assembly described above eliminates the need for packing supports in contact with the surfaces to be silvered, and avoids the possibility of contamination of these surfaces by the supports (such as asbestos tape or corrugated paper).

SEALING-OFF

When the silvered flask has been baked and evacuated to the highest possible vacuum, it must be sealed off at the prepared constriction. If, because of the design of the baking oven or of the vacuum system, it is inconvenient to make the seal at the required place, an alternative procedure can be employed. A second constriction is made in the pump stem, in an accessible position. The first constriction can now be enclosed in the oven and will be heated there and outgassed. The flask is sealed off at the second constriction and removed from the oven; the short remaining length of pump stem can then be carefully sealed off and the seal flame-annealed.

Cartesian Manostat

The Cartesian manostat is a device used to maintain the pressure in a partially evacuated sytem to within about 1 % of some previously determined value.

The theory governing the functioning of Cartesian diver manostats, and a number of modifications of the device, are dealt with by Gilmot.[4]

The method of constructing a simple type is set out below; see also Fig. 9.2.

The float is made from 18 mm tubing of the thinnest available wall-thickness. One end is constricted so that a small rubber stopper will fit the opening tightly. This end is now cut off straight and fire-polished.

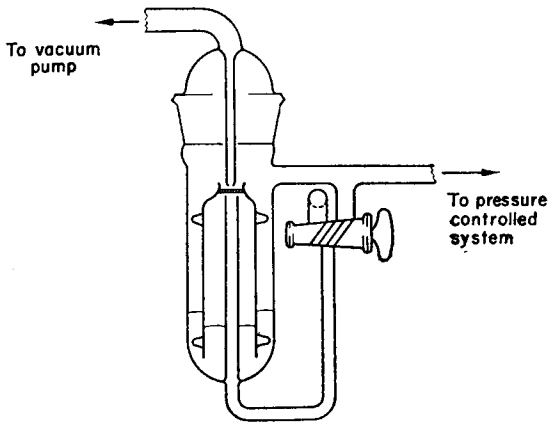

FIG. 9.2. A Cartesian manostat.

Three small glass points are drawn out near the shoulder and uniformly spaced round the circumference. The length of these projections is adjusted so that the float will just enter the jacket with only two projections touching the inside wall.

The float is now cut to give a total length of about 16 cm. The cut end is fire-polished and three small points drawn out near the end and similar to those at the shoulder.

The jacket is made from normal wall tube of 29 to 30 mm bore. A B34 standard ground-glass socket is joined to one end, and a 5 mm tube 16·5 cm long is internally sealed to the other end. The length of the jacket, including the socket, should be about 22 cm. A 30 cm length of 8 to 9 mm heavy-wall tube is joined to the ring seal, and a 15 cm length of the same tube joined to the jacket just below the socket.

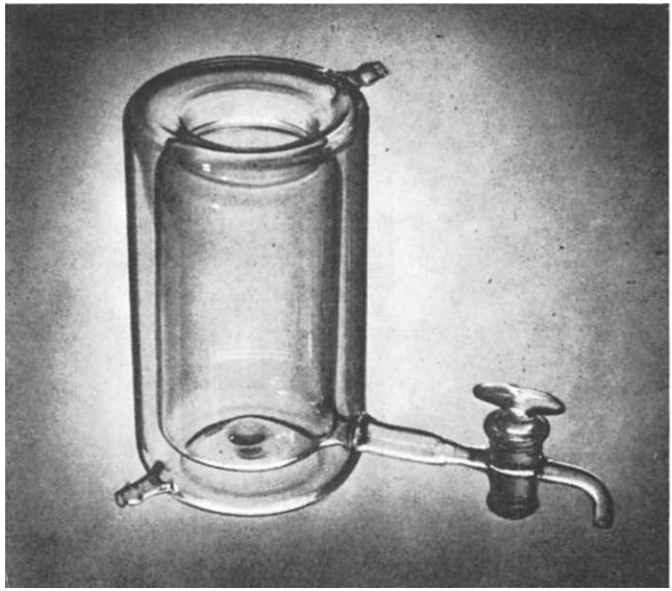

PLATE 9.1. A jacketed beaker. Made in the lathe.

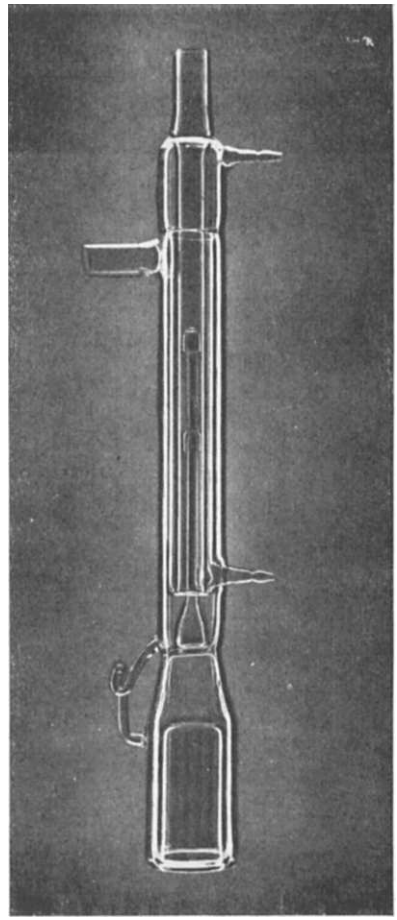

PLATE 9.2. A mercury diffusion pump.

A double oblique-bore stopcock is now joined to the jacket side tube by one of the adjacent side-arms, the second arm being cut off 2 cm from the stopcock barrel, the third side-arm is cut at about 3 cm from the barrel. The tube attached to the bottom of the jacket is bent round till it is in the same plane as the jacket, side tube and stopcock. It is then cut off at a suitable length and gently adjusted so that the open end is close to the stopcock side-arm and exactly in line with it. These two ends are now joined together, using a thin rod to fill up the gap.

A Mercury Diffusion Pump

This pump is shown in Fig. 9.3 and Plate 9.2. The sketch should be studied, the diameters of the tubing for the components should be noted and the glass selected, cleaned and set aside.

The condenser is made as follows: A 26 cm length of 23 mm outside diameter borosilicate glass is ring-sealed into a 30 cm length of 30 mm outside diameter tube. The end of the inner tube remote from the ring-seal must be stoppered to permit effective blowing. A corrugated-paper sleeve supports the inner tube. The water-inlet tube is now attached to the outer tube close to the ring seal. The outer tube is now cut off at 21 cm from the ring seal. The cut end is flanged out so that the flange just enters the jacket. The open end of the inner tube is similarly flanged. The water-inlet tube is cut off and tooled to such a length and shape that it touches the jacket when the condenser is concentric with the jacket. Should this water-inlet tube be too short, some difficulty will be experienced in making a neat seal to the jacket. Should it be too long the condenser will be asymmetric and subsequent steps will give unsatisfactory results.

The internal seal at the top of the condenser is now made and the gas-inlet tube and water-outlet tube attached. The second internal seal is made without delay and the backing pump connecting-tube joined to the jacket. This end of the condenser is flame-annealed and set aside to cool.

The boiler is made by ring sealing the inner and outer tubes

together and attaching a short length of 6 mm tubing to serve as part of the mercury return line.

The mercury jets must be made accurately so that the annular space for the vapour is 1 mm wide or as near to 1 mm as the available tubing permits.

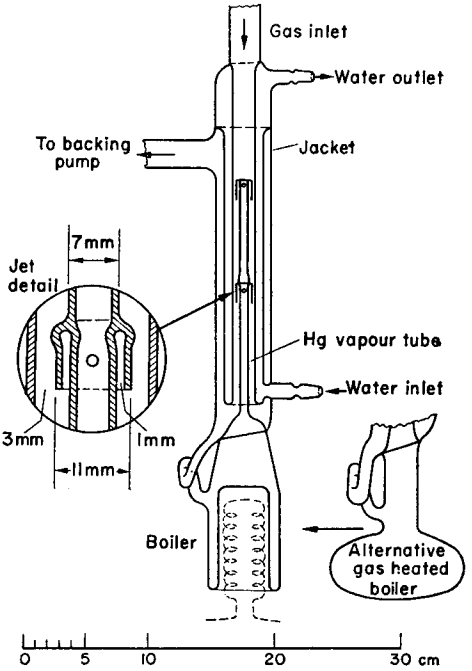

FIG. 9.3. A mercury diffusion pump and an alternative boiler for gas heating.

The annular space between the outside of the jet umbrellas and the inner surface of the condenser should be 3 mm wide.

The jets are made separately and care taken to make the mercury vapour holes neatly. The umbrellas are cut to length after the internal seals are made. These cuts should be straight; they should not be fire-polished.

The jets are joined together, just above the lower one, and a slight constriction made in the join. This constriction ensures that adequate mercury vapour is diverted from the main upward stream to make the lower jet function. It is essential that the jets be in alignment.

A short length of 23 mm tubing is attached to the mercury vapour tube, cut off at a slight angle to the horizontal and flanged out in preparation for sealing to the outer jacket.

The outer jacket of the condenser and the boiler are now cut to length. The mercury vapour tube and jets are supported in the centre of the condenser with corrugated paper, or with a suitable number of layers of ordinary paper.

The support must keep the jets in a central position and prevent them from moving along the length of the condenser. It must be capable of being withdrawn when the pump is completed, otherwise it can be removed with the aid of dilute nitric acid.

The next step is to make the water inlet seal and attach the water-inlet tube.

The boiler is now joined to the jacket, the mercury return tube must be in line with the lowest part of the vapour tube flange, and on the opposite side of the pump to the water-inlet tube. Without any delay the vapour tube flange is sealed to the jacket and the prepared mercury return tube joined to the jacket, just above the internal seal. All the worked glass in this region is now flame-annealed.

The mercury return tubes are cut to length and adjusted so that they are in line and almost touching.

A handlamp can be used to make the adjustments and also to join up the ends.

When the paper sleeve has been removed the pump should be oven-annealed. It is essential to avoid overheating the glass during annealing, especially if the pump lies flat in the oven, as this may result in the vapour tube bending slightly and displacing the jets from their central position.

When filling the boiler with mercury, remember to completely cover the inner tube as the quantity of vapour generated per unit area depends on the area of exposed mercury surface.

The type of boiler fitted to this pump is designed for electric heating. Should an internal heater be available the boiler must be designed to accommodate it.

An external heater can be made by winding a length of flat nichrome strip round the boiler, securing the ends with brass bands fitted with terminals and enclosing the heater in a layer of asbestos cloth. Sodium silicate is sometimes used to enclose the nichrome strip; however, it attacks the glass and is therefore undesirable.

Should gas heating be preferred, the boiler should be made an alternative shape shown in Fig. 9.3.

It is worth noting that little, if any, improvement in pumping speed is obtained by allowing the mercury to boil vigorously. Very gentle boiling is all that is necessary.

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CHAPTER 10

Vacuum Technique

THE atmosphere which surrounds us consists of about 20% oxygen, 77% nitrogen, 1% argon and other gases, and 0·1% carbon dioxide, with small variations in these proportions depending on locality. There is also present varying amounts of water vapour, usually between 1% and 2%.

The pressure of the atmosphere depends on altitude: at sea-level it is close to 760 mm of mercury (mm Hg) and falls almost to zero at 1125 km (700 miles) above the equator.

In some scientific work it is desirable to reduce the pressure in the apparatus used to some value below atmospheric pressure. The process used to reduce the pressure in any enclosed space is known as evacuation. The space, when evacuated, is known as a vacuum.

Although, in dictionary terms, a vacuum is defined as a space entirely devoid of matter, it must be appreciated that such a state of affairs is very difficult to achieve. In fact it is very nearly impossible to remove the last traces of gas or vapour from any enclosed space, since this would involve reducing the temperature of the confining vessel to absolute zero (−273 °C).

The evacuation of vessels is an important process and has many applications in teaching, research and industry.

Although engineers and physicists make extensive use of metal systems, which are outside the scope of a glassblower, glass vacuum systems are widely used in chemistry laboratories for many short-term research projects and for teaching and demonstration purposes.

An efficient and serviceable vacuum system can be quickly made by a reasonably competent glassblower. Leaks in the

glassware are easily located and repaired. Modifications and repairs to the vacuum line can be carried out with little delay.

In general, research workers are concerned with the theoretical aspects of their particular problems rather than with the details of the design and proper functioning of the apparatus required to help them solve the problems.

Since in the course of a few years a glassblower may be called upon to construct a number of vacuum systems to serve various purposes, it follows that he can become, in a modest way, an authority on vacuum line design and layout, and expert in vacuum technique.

Experience in these aspects of the work must be supported by elementary theoretical knowledge of the behaviour of gases at low pressures, and of the functioning and limitations of the different types of vacuum pumps. It is almost essential that the glassblower know something about mercury as a pressure gauge liquid and as a diffusion pump liquid. He should be familiar with vacuum stopcock greases and pump oils, with refrigerants and cold traps, with pressure-measuring devices and their limitations. He should be aware of the importance of good quality stopcocks and interchangeable ground glass joints, of the techniques used to lubricate them, and of their function in a vacuum line.

Let us consider, first, the units in which gas pressures, of less than one atmosphere, are measured: Torricelli, an Italian experimenter, established that the pressure of the atmosphere would support a column of mercury 760 mm high. Standard atmospheric pressure is therefore said to be 760 torr or 760 mm Hg.

$$1 \text{ mm Hg} = 1 \text{ torr}$$

$$0{\cdot}1 \text{ mm Hg} = 1 \times 10^{-1} \text{ torr.}$$

The micron, 1×10^{-6} m, is sometimes used.

$$1\,\mu \text{ Hg} = 1 \times 10^{-3} \text{ torr.}$$

Let us now consider the flow of gas in a tube. The molecules of a substance in the liquid or gaseous state are in rapid motion,

and in the gaseous state the number of particles in unit volume is much smaller than in either the solid or liquid states. Intermolecular attractive forces are consequently much reduced, and a mass of gas in an enclosed space will diffuse in all directions to fill the entire system.

The number of molecules in 1 cm^3 of gas at STP (standard temperature, 0°C; standard pressure, 760 torr) is $2{\cdot}69 \times 10^{19}$. All these molecules are in random motion but because of collisions between them the direction of motion changes frequently. The average distance traversed by a molecule between collisions is termed the mean free path.

The type of flow of a fluid through a tube depends on the rate of flow. At low speeds it is laminar, where all the particles are moving parallel to each other as in a gently flowing stream. As the speed increases a point is reached when the flow is turbulent. The point at which this occurs is determined by the shape of the containing vessel or tube. The above discussion refers to a fluid, where collisions between the particles themselves are more important than collisions between the particles and the vessel or tube walls, and applies to a gas where the mean free path is small compared with the tube diameter. As the gas pressure is reduced and the mean free path becomes comparable with the tube diameter, a third type of flow becomes important. This type of flow, known as a molecular flow, is significant in vacuum systems where the pressure is about 1 torr.

TABLE 5

Pressure (torr)	760	10^{-1}	10^{-3}	10^{-6}
Mean free path (cm)	10^{-5}	5×10^{-2}	5	5000

The average diameter of a gaseous particle is about 3×10^{-8}cm, which is, as will be shown, very small compared with the mean free path. Table 5 gives the mean free path for air at some pressures.

Thus at pressures below 10^{-3} torr it is apparent that collisions between the gas molecules will be much less frequent than colli-

sions between them and the wall of a tube, less than 5 cm diameter.

The flow of gas in a vacuum system is of all three types successively when the pressure is being reduced from atmospheric to 1 torr or less. Turbulent flow occurs for a short time at the beginning of the evacuation when the gas velocity is high. Laminar flow takes place when the mean free path is small compared with the tube diameter; under these conditions the gases near the tube wall are almost stationary, and those near the centre of the tube have the maximum velocity. As the pressure is reduced to about 1 torr, molecular flow is first established (Fig. 10.1).

It should be noted that, at pressures below 1 torr, the flow of gas along a tube is the result of random motion of the molecules rather than of pressure difference between the ends of the tube, and that the rate of gas flow is much reduced if the tube is long or of small diameter.

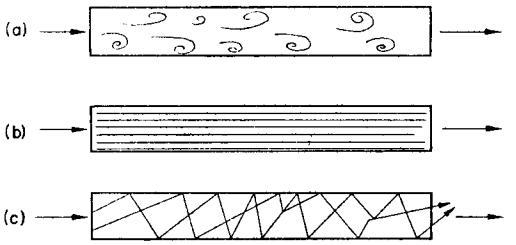

FIG. 10.1. (a) Turbulent flow, (b) laminar flow, and (c) molecular flow of gas in a tube.

A simple experiment, employing glass tubes of various lengths and diameters which connect, in turn, a 5000 ml flask to a diffusion pump, will now be described.

The apparatus was set up as shown in Fig. 10.2, first with the flask connected directly to the diffusion pump by B24 ground-glass joints, and then connected by each of five tubes, with different dimensions, in turn.

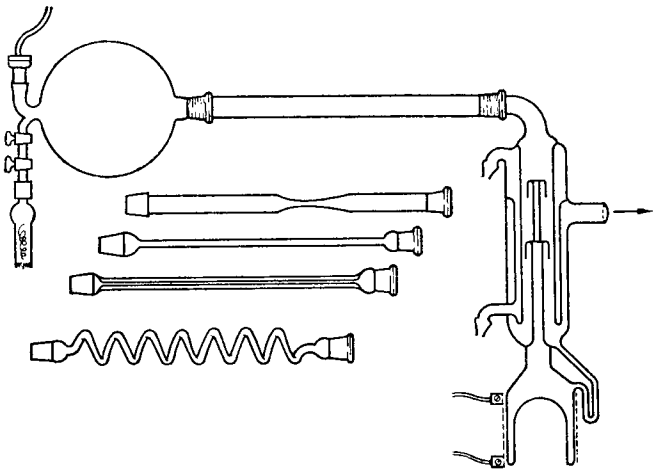

FIG. 10.2. Glassware used in an experiment designed to show the effect of tube diameter on pumping speed.

In each case the system was pumped down to 10^{-3} torr, a small quantity of dried air was admitted and the time taken for the pressure gauge needle to move from 10^{-2} torr to 10^{-3} torr was noted.

The results are set in Table 6.

The pressures were measured on a thermocouple gauge calibrated with a McLeod gauge, using dry air. (Pressure gauges and diffusion pumps will be discussed later in this chapter.)

TABLE 6

5000 ml flask only	14·1 sec
Connecting tube	
20 mm bore 50 cm long	21·5 sec
20 mm bore 50 cm long with a short constriction 7·3 mm bore	30·8 sec
10 mm bore 50 cm long	64 sec
3 mm bore 50 cm long	1800 sec
3 mm bore 150 cm long	10,800 sec

These results show that in any vacuum system the tubes in the vacuum line must be kept as large in diameter as possible and as short in length as convenient or the rate of evacuation will be reduced.

In addition to the above results it was found that the pressure in the flask was eventually reduced to 10^{-5} torr in all cases except the last. With the 3 mm bore tube, 150 cm long, the ultimate vacuum did not fall much below 10^{-3} torr after 4 hr pumping.

Types of Pumps

The ultimate vacuum required in any system determines the type of pump, or pumps, that should be used.

WATER-JET PUMP

A water-jet pump, sometimes known as an aspirator pump or filter pump, will reduce the pressure in a closed system to within the range 760 torr to 20 torr. The pressure can be maintained at any desired value by including a manostat in the vacuum line. Water-jet pumps should only be used where there is a plentiful supply of water at an average pressure of 1 kg/cm^2 (15 lb/in^2) with a minimum pressure of 0·5 kg/cm^2 (8 lb/in^2).

This type of pump is widely used in laboratories as an aid to filtration, for the partial evacuation of desiccators and for reducing the pressure in distillation apparatus. Such pumps are all too frequently connected directly to the apparatus to be evacuated and while this procedure is effective it must be under constant supervision. There are, however, many and varied demands on any laboratory water supply, so that the pressure may fluctuate and fall below the safe pressure for the pump, in which case the water will flow into the evacuated space to spoil the work in hand.

A non-return valve fitted to the pump inlet will reduce this hazard. A Buchner flask fitted with a rubber stopper carrying a

vacuum release stopcock and a glass tube reaching to the bottom of the flask will provide a means of preventing a back flow of water into the evacuated system. Should the back flow of water be insufficient to close the non-return valve, then it will be seen to enter the Buchner flask. The stopcock can be opened very slightly, the resulting increase of pressure in the flask will allow the pump to remove the accumulating water and so prevent flooding of the apparatus (see Fig. 10.3).

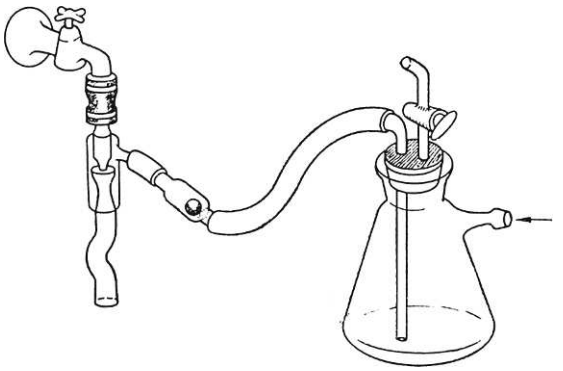

FIG. 10.3. A water-jet pump fitted with safety devices.

It should be noted that the pressure difference between the outside and the inside of a system evacuated to 20 torr is approximately

$$13{\cdot}5(76 - 2) = 1000 \text{ g/cm}^2.$$

The pressure difference due to an internal pressure of the order of $1.\times 10^{-6}$ torr is approximately

$$13{\cdot}5(76 - 1 \times 10^{-7}) = 1026 \text{ g/cm}^2.$$

The increase in pressure is about 26 g/cm^2 or less than 3%.

It follows that the hazards normally associated with high vacuum apparatus are also present, and to approximately the same degree, with vacuum pressures of about 20 torr. The glassblower should wear his safety spectacles when working

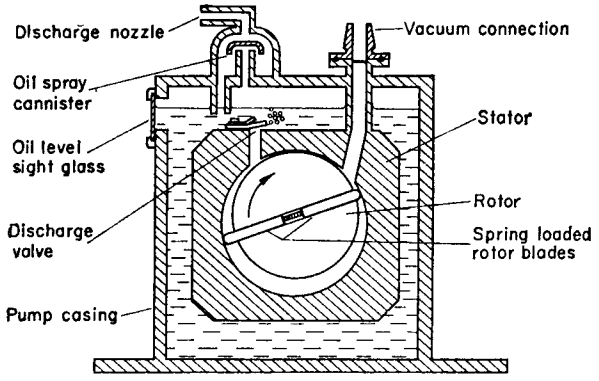

FIG. 10.4. Section through a rotary oil pump.

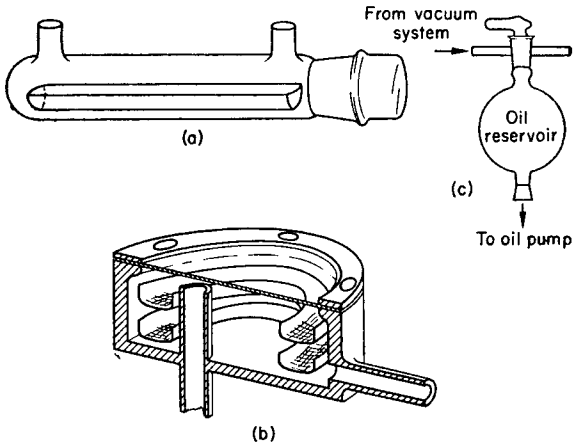

FIG. 10.5. (a) A glass moisture trap containing a boat for phosphorus pentoxide. (b) A metal moisture trap in section, showing the trays for phosphorus pentoxide. (c) Oil reservoir, fitted to the vacuum connection on a rotary oil pump. The two-way stopcock is opened to the atmosphere when the pump is stopped.

with any type of glass vacuum system. He must not include glass vessels of even moderate capacity, if they have flat bottoms, unless they are of exceptional wall-thickness, and are designed, like Buchner flasks, to withstand high pressure differences. Flat-bottomed boiling flasks and Erlenmeyer flasks must never be evacuated, as such vessels will collapse with an accompanying implosion.

Rotary oil pumps

A single-stage rotary oil pump should be used to cover the range 20 torr to 1×10^{-2} torr. Such pumps are enclosed in a cast metal case which is filled with a special oil. This oil has a low vapour pressure, and must be protected from contamination by water and other solvents. A phosphorus pentoxide (P_2O_5) trap will remove water vapour, but a refrigerated trap is necessary if solvent vapours are present. Figure 10.4 shows a rotary oil pump in section and Fig. 10.5 shows a glass and a metal P_2O_5 moisture trap.

Two-stage rotary oil pump

If pressures within the range 1×10^{-2} torr to 1×10^{-4} torr are required, a two-stage rotary oil pump must be used. This type of pump is simply two single-stage pumps arranged in tandem, one backing the other, but both enclosed in the same oil bath and driven by a common shaft. As with single-stage pumps the oil must be protected from contamination. Some modern rotary oil pumps are equipped with an air ballast attachment; this is essentially a small adjustable air leak into the pump cylinder near the outlet. This small flow of air assists in clearing the oil of vapours, and if properly used, keeps the vapour pressure of the oil low. However, the quantity of vapour handled by the pump must be kept to a minimum as the effect of air ballast is slow.

The performance of these pumps depends on the condition of the oil. Should this be contaminated to such an extent that

the ultimate pressure is considerably above that previously obtained, then the oil should be drained off, the pump flushed out with new oil and then refilled with new oil.

It is worth noting that vacuum pump oil of the recommended quality should be used. Any other oil used in the pump will result in a poor ultimate vacuum.

When pumps of this type are stopped then air must be admitted to the pump inlet. Should this step in vacuum procedure be overlooked, the oil will seep through the lubricating channels and be forced into the vacuum system by atmospheric pressure, flooding the traps, pressure gauges and all the evacuated space. Pump manufacturers supply a non-return valve which floats on the ascending oil to make a seal confining the oil to the pump body. These valves are generally effective, but can be put out of action by small fragments of foreign matter in the oil finding their way into the valve seal. An oil reservoir of appropriate capacity, fitted with a two-way stopcock (Fig. 10.5) is a much more reliable safeguard. Much time can be lost cleaning out a pump system which has been flooded with oil.

Diffusion pump

Should a pumping system be required to achieve pressures within the range 10^{-2} torr to 10^{-6} torr a more elaborate system must be used. A basic design for such a system is shown in Fig. 10.6.

A rotary oil pump, capable of maintaining the pressure in the system at 1 torr or less, is essential. The oil pump is connected, through a trap tube, to the high pressure side of the mercury diffusion pump. The low pressure side of the diffusion pump leads to a short manifold through another trap designed to be surrounded by a refrigerant. The manifold is fitted with side tubes for a pressure gauge, for a vacuum release stopcock and for a main stopcock. The layout beyond the main stopcock depends on the nature of the project, and may be a direct connection to a vacuum jacket under preparation, to an elaborate manifold used for radioactive investigations or to a fractionating still operating under very low pressure.

A mercury diffusion pump operates in quite a simple manner. Mercury vapour from the boiler (Fig. 9.3) ascends the innermost tube and emerges from the jets at high velocity. Provided that

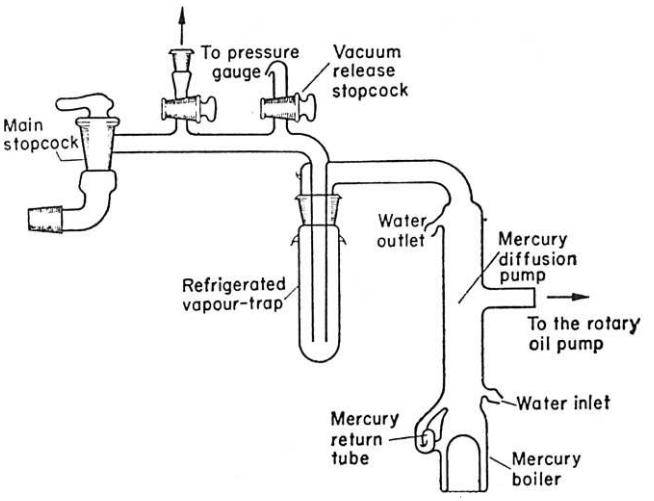

FIG. 10.6. A simple high vacuum system. The diffusion pump is connected to the rotary oil pump through the oil reservoir. The apparatus to be evacuated is attached to the standard joint below the main stopcock.

the pressure in the diffusion pump is such that the gas flow is molecular (less than 1 torr), the gases in the vicinity of the rapidly moving mercury stream diffuse into the mercury vapour and are knocked downwards. The mercury condenses on the water-cooled surfaces and returns to the boiler. The gases in the region just below the vapour stream are unlikely to diffuse upwards past the barrier of high velocity mercury vapour. The gases move from the connecting tubes and the apparatus into the pump by molecular flow and are continuously removed.

It must be stressed that this type of pump can function with reasonable efficiency only when the pressure is at, or below, 1 torr and molecular flow is possible.

The boiling point of mercury at atmospheric pressure is 356·9 °C, and at 1 torr it is about 200 °C. It follows that the mercury diffusion pump boiler should not be heated until the

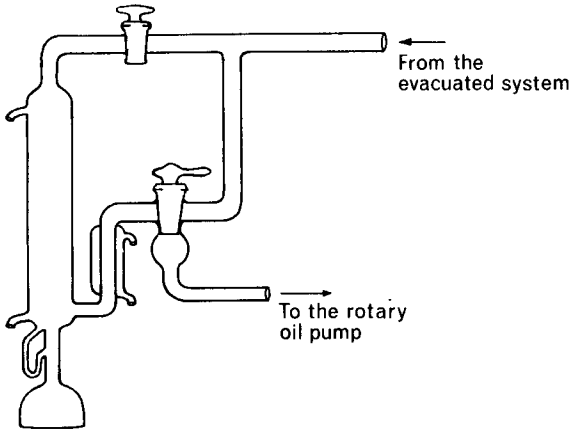

FIG. 10.7. Stopcock arrangement for by-passing a diffusion pump. Such an arrangement allows the rotary oil pump to recover the low pressure required before the diffusion pump can operate and eliminates the need to discontinue the supply of heat to the pump boiler. The diffusion pump can be made to function again by opening the stopcock and turning stopcock handle through 180°.

pressure is reduced to 1 torr. Should the pressure be allowed to rise considerably, for any reason, the heating should be discontinued. Otherwise, when the pressure falls the mercury will be superheated and may bump violently. Mercury is a dense liquid, 13·55 g/cm^3 at 0 °C, and large fast moving quantities of it may break the delicate jets or the boiler.

Should large masses of air or other gases pass through the diffusion pump when the mercury vapour is flowing freely, the vapour will be swept past the condensing surfaces into the backing pump and its connections.

The vapour pressure of mercury at room temperature is about 1×10^{-3} torr and this will be the limit of the pressure that can be reached unless a refrigerated trap is used to condense the

vapour. Without such a trap the mercury vapour will diffuse throughout the vacuum system.

When a pumping system with a diffusion pump is used in such manner that the cycle of operations results in pressure variations which, at times, rise considerably above 1 torr the diffusion pump can be by-passed by an arrangement of stopcocks. The pressure in the diffusion pump can therefore be maintained at 1 torr or less and the backing pump used to recover low pressure in the system. With such an arrangement the diffusion pump heating need not be interrupted. A method of by-passing the diffusion pump is shown in Fig. 10.7, and a more elaborate arrangement in Fig. 10.8.

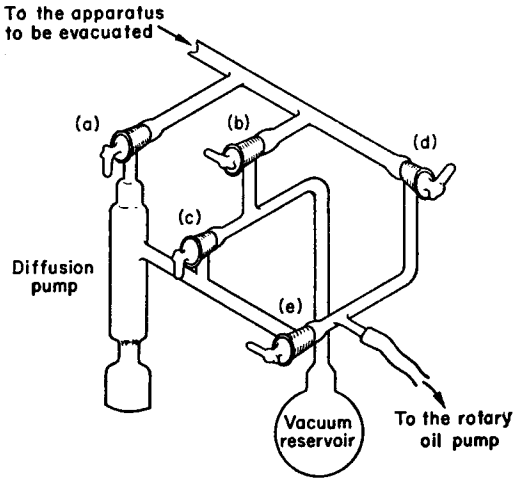

FIG. 10.8. This stopcock arrangement permits the following operations. (1) By closing all the stopcocks except (d) the diffusion pump is by-passed. (2) When (d) and (b) are open the rotary oil pump will roughly evacuate the vacuum reservoir. (3) With (c) and (d) closed and (a), (b) and (e) open the diffusion pump will evacuate the vacuum reservoir. (4) To use the evacuated reservoir as a backing volume. Close (b), (d), and (e) and open (a) and (c). The rotary oil pump is then isolated and switched off. This arrangement is useful for prolonged pumping at low pressures; it should not be used until all the glassware in the system is thoroughly degassed.

PUMPING SPEED

The pumping speed of a rotary oil pump is a measure of the volume swept out per minute by the vanes. Small pumps, of the type used in laboratory work, have a speed of about 22 l./min. Large pumps, such as are used for many industrial purposes, have a speed of 250 l./min, measured at normal pressure.

The speed of simple mercury diffusion pumps is about 10 l./sec; this means that under optimum conditions (i.e. suitable backing pump and minimum resistance to gas flow on the high vacuum side of the pump), 10 l. of gas at 10^{-6} torr will be removed per second. The speed of a diffusion pump connected to a vacuum line is always less than the rated speed. The reduction in speed is least when the pump is directly attached to a system, such as a bell jar; the inclusion of long- or small-bore connections can reduce the effective speed to 1 %, or even less, of the rated speed.

The effective speed of a diffusion pump also depends on the capacity of the backing pump. The following figures[1] are given as a guide to the selection of matching pumps.

Mercury diffusion pump speed (l./sec)	3	6	40	300
Rotary oil pump speed (l./min)	20	20	50	210

REFRIGERANTS

The best refrigerant is LN, liquid nitrogen, which has a temperature of about −196°C. LN must be transported and stored in insulated or vacuum-jacketed containers. It may be purchased from nearby industrial gas companies, but by far the most convenient source is a LN plant installed in the laboratory building.

Liquid air is potentially dangerous and is now rarely used. Liquid oxygen should never be used as a refrigerant, it can cause an explosion when in contact with some organic substances.

Should LN be poured into a dewar flask which contains water, no matter what its source, then the ice which forms will break the dewar. All dewars must be dried out before adding LN.

If a flow of air or oxygen is allowed to pass through a refrigerated trap surrounded by LN of recent manufacture, then oxygen may be condensed within the trap. If the system is then isolated from the pump and left overnight, the remaining refrigerant will boil off and the oxygen in the trap will evaporate. The resulting increase in pressure can be enough to blow the key out of some types of stopcock. Other condensed vapours will also evaporate when the refrigerant is removed and will pass into and contaminate the rotary pump oil when the apparatus is next used. Vacuum apparatus used by successive groups of students suffers most from this indifferent technique. The students should be instructed to clean out all refrigerated traps while they are cold.

LN is normally stored in a vacuum-jacketed metal container, with provision for easy tipping to dispense the contents. The outlets should be provided with a dust cover, but must not be tightly closed, as dangerously high pressures will develop through evaporation. LN dispensers are shown in Fig. 10.9.

Solid CO_2 dissolved in acetone has a temperature of about $-80°C$; this is adequate for many purposes and much cheaper

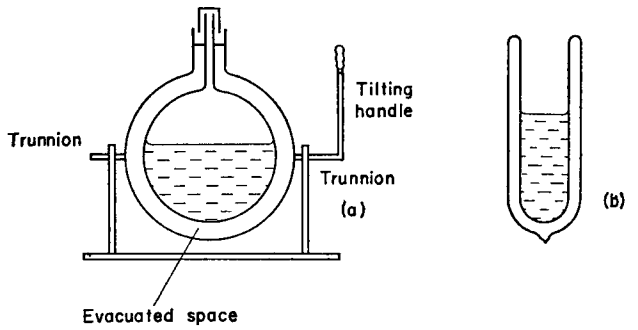

FIG. 10.9. A LN dispenser (a) mounted on a stand and capable of being tilted to fill small capacity Dewar flasks. This type of dispenser has a capacity of about 50 l., and is made of metal. A Dewar flask (b). This is a double-walled glass vessel, the inner space is evacuated and its glass surfaces are coated with silver. Capacity range up to 1·5 l.

than LN. The CO_2 in solution evaporates fairly rapidly and should be replaced by adding small pieces to the Dewar flask. The usual procedure is to have some solid CO_2 floating in the solution at all times.

Vacuum Gauges

The ultimate pressure required in any vacuum system determines the type or types of gauge that should be used.

Open limb U-tube manometer

An open limb U-tube, 85 cm high, made from tubing about 10 mm bore and having a short 1 mm bore constriction in or near the bend, can be used for approximate measurements of pressure within the range 1500 mm to 20 mm Hg. The constriction in the tube reduces surging of the manometric liquid, usually mercury, when the pressure changes and damps out oscillation of the liquid column.

The difference in level of the mercury surfaces in the limbs, as measured with a metre stick, gives the pressure in the limb attached to the apparatus. The open limb is exposed to atmospheric pressure, which must be measured on the laboratory barometer and allowed for. A good standard of accuracy in measuring pressures is possible with this type of gauge if the mercury and the inside surface of the glass U-tube are clean and the levels are measured with a cathetometer.

A cathetometer is a telescope fitted with cross-wires and mounted on an adjustable carriage moving over a vertical scale and carrying a vernier.

Since the cohesive forces between the molecules of mercury are greater than the adhesive forces between the mercury and the glass, the level of the mercury surface in a tube is depressed. The amount of depression depends on the cleanliness of the mercury and glass, and also on the bore of the tube. Where accuracy is required in measuring a pressure by taking the difference

in surface levels of a manometric liquid in any pressure measuring device, these levels must be measured in tubes of exactly the same bore. Otherwise some correction for depression or elevation must be made, and the measurement will then be complicated and the errors increased.

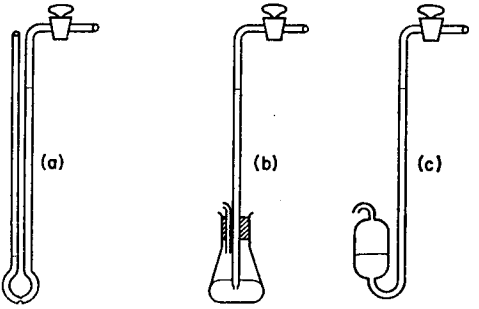

FIG. 10.10. Three types of open limb U-tube manometer.

Perfect cleanliness of mercury and glass is difficult to maintain. The glass tube, near the mercury surface, should be lightly tapped with a pencil just before the readings are taken, as even slight contamination of the surfaces will tend to make the mercury adhere to the glass and give a false reading.

An open limb U-tube is shown in Fig. 10.10 together with a number of variations in design.

CLOSED LIMB U-TUBE

The closed limb of this manometer is evacuated and measurements are independent of atmospheric pressure. The height of the U-tube determines the range. This manometer has many uses: for instance, a short type will show at a glance if the pressure in a vacuum system is low enough to allow the diffusion pump to be brought into use.

Two simple types of closed limb manometers are shown in Fig. 10.11. The closed limb in (a) is evacuated during the

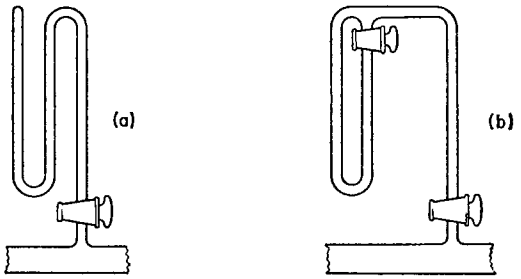

FIG. 10.11. Two closed limb manometers.

construction of the gauge. Should a pressure of gas or vapour accumulate, for any reason, in the closed limb, then the gauge must be detached from the system for servicing. Using a carefully constructed and properly treated gauge there should be no need for such servicing. The author has one which was made 10 years ago and has never been cleaned or re-evacuated.

Type (b), fitted with a stopcock at the top of the closed limb, is suitable for low vapour pressure manometric oils and may be re-evacuated at any time. The oil wets the glass and some time should be allowed for drainage before measurements are taken. Further, the oil tends to creep along the glass wall into the system, where it may be undesirable. Manometers of type (b) (Fig. 10.11) may be used with advantage in measuring the pressure in gas discharge tubes. The relative density of low vapour pressure manometric oils is less than 1·0, so pressures of about 20 torr will be indicated as a difference in surface level of about 27 cm. Such gas pressures can be read directly and adjusted accurately with the aid of this type of gauge.

THE MCLEOD GAUGE

This gauge is used to measure low pressures within the range 10 torr to 1×10^{-5} torr. It is assumed that dry gases at low pressures obey Boyle's law, which states that the pressure of a

given mass of gas, kept at constant temperature, is inversely proportional to its volume.

In symbols:

$$p = 1/vk$$

or

$$pv = \text{a constant.}$$

The gauge is shown in its simplest form in Fig. 10.12. To measure the gas pressure in a system to which the gauge is attached the reservoir is lowered so that the mercury level in the gauge falls below the opening in the bottom of the bulb *A*. The pressure in this bulb will assume the pressure in the system in about 30 sec. The reservoir is now slowly raised and the increase in height of the mercury in the gauge will isolate a volume of gas in the bulb and compress it into the capillary tube *B*.

The reservoir level is adjusted so that the surface of the mercury in capillary *C* is exactly at the zero mark. The difference in level of the mercury in tubes *B* and *C* is read on the calibrated scale and gives the gas pressure in the system in mm Hg or torr.

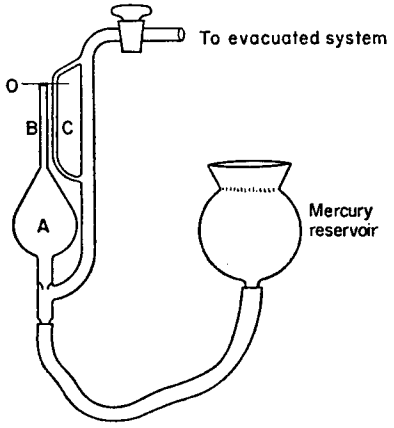

FIG. 10.12. A McLeod gauge. The gauge head and reservoir are made of glass. The range of pressure that can be measured depends on the volume of bulb *A*, and the bore and length of the capillary tube *B*. Capillary tubes *B* and *C* are made from tubing of identical bore to eliminate the need for capillary corrections.

In calibrating the scale it is essential to measure the total capacity of bulb A and capillary tube B, it is also necessary to measure accurately the cross-sectional area of B and C and to make them from the same tube or from tubes of exactly similar bore. The latter step eliminates the need for capillarity corrections.

Let V mm^3 be the capacity of $A + B$, v mm^3 be the capacity per mm length of capillaries B and C and h mm be the difference in mercury level in B and C. Then if p mm is the required pressure, we have from Boyle's law

$$pV = (p + h)\, vh.$$

Now p is usually very small compared with h (in a common type of McLeod gauge p is 1×10^{-5} torr when h is 1 mm), so we may write

$$pV = h^2 v$$

or

$$p = v/V h^2.$$

Since v and V are both known for each gauge we have

$$p = h^2 \times \text{a constant.}$$

The McLeod gauge will measure the pressure of dry gas to an accuracy of about 1% and is used to calibrate other types of vacuum gauges. It is slow in operation and does not give continuous readings. It does not measure the pressure accurately if condensable vapours are present, and is limited to 10^{-3} torr if no refrigerated trap is used.

In its original form this gauge must have a stand not less than 150 cm high, it is therefore unsuitable for bench assemblies. A number of modifications, designed to make the instrument more compact, are shown in Fig. 10.13. In the short form the mercury is raised by admitting air to the evacuated reservoir and lowered by re-evacuating the reservoir. The rotary oil pump may be used for this re-evacuation if provision is made for isolating the pump from the system. Otherwise air pumped from the reservoir will also flow into the evacuated space and temporarily raise the pressure. Alternatively, a source of rough vacuum, such

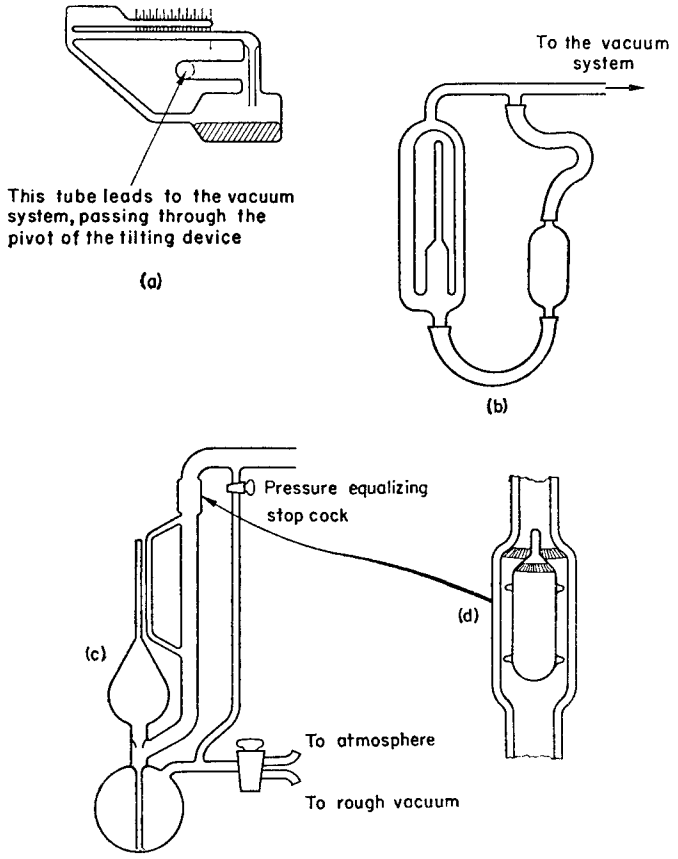

FIG. 10.13. (a) A tilting McLeod gauge shown in the horizontal position. To measure a low pressure the gauge is tilted through 90°, on its stand; the mercury flows into and seals off the gas in the small bulb. This gas is compressed into the capillary tube and the pressure is read off on the calibrated scale. The gauge should be returned to its original position. (b) Is an ingenious and compact design due to Lloyd. (c) A short form of McLeod[2] gauge fitted with a pressure-equalizing stopcock and a glass float valve shown in detail in (d).

as a water-jet pump, may be used. This is often the preferred method when the pressure sought is close to the ultimate performance of the pump.

The tilting gauge is compact and does not require much mercury. It is reasonably accurate over the range 10 torr to 10^{-3} torr provided the volume of mercury is correct. The gauge must be kept in its horizontal position except when a pressure is being measured; it is then tilted through 90°. The mercury flows under gravity and encloses a fixed volume of gas which is then compressed into the closed capillary tube. The calibration and reading are the same as for an ordinary McLeod gauge.

The technique of using the tilting gauge is straightforward and easily learned, that of the short form of McLeod gauge is complicated by the fact that the pressures in the gauge and in the reservoir must be kept nearly equal. A simple modification has been used by R. F. Gledhill of Victoria University, Wellington, New Zealand. It consists of a tube connecting the reservoir to the vacuum system and fitted with a stopcock to allow the pressures to be equalized without the possibility of violent surging of the mercury during the initial evacuation (see Fig. 10.13).

Should this type of gauge be left evacuated for some time, and should air leak into the reservoir for any reason, the increase in pressure will raise the level of the mercury in the gauge and it may enter the vacuum line. A glass float valve is sometimes fitted above the gauge. Such float valves are effective if they have a properly ground surface making good contact with a suitably ground seat. They must not, however, be raised quickly, or the momentum of the mercury will break either the float or its jacket (Fig. 10.13).

Other vacuum gauges

A Bourdon type dial gauge may be used to indicate pressures within the range 20 torr to 1 torr with an accuracy of about $\pm 3\%$. A bellows-operated gauge magnifies the movement of a metal bellows by an optical method and indicates pressure

difference over the range 50 torr to 10^{-2} torr. Neither bellows gauges nor Bourdon gauges are in common use in laboratory work.

High frequency leak tester[3]

This instrument is almost essential where glass vacuum systems are made or used. The high frequency discharge from the electrode will detect very small pinholes in glass joins, which are almost invisible to the naked eye. The discharge concentrates as a white streamer between the electrode and the pinhole or crack, and is easily seen in a darkened room. The electrode discharge ionizes the low pressure gases in a glass vacuum system, and the pressure in the system can be estimated from the colour and nature of this ionized gas. Air at about 10^{-1} torr is mauve in colour. As the pressure falls this colour fades. At about 10^{-2} torr a patch of fluorescence appears on the inside wall of the tube, opposite the electrode. This fluorescence disappears at pressures below 10^{-4} torr.

Spectrally pure gases give characteristic colours:

Helium	Bright yellow–white
Argon	Violet
Neon	Red

These colours are usually masked by the blue-grey of water vapour.

Mercury vapour, at room temperature, gives a blue colour, and at higher temperatures, a bright green colour. A persistent mauve (or air) colour, usually indicates some kind of leak, which should be sought out and corrected before proceeding further with the work in hand.

Thermal gauge, or Pirani gauge

This popular electrical vacuum gauge covers the range 10 to 10^{-4} torr. The gauge consists of a heated filament of some metal with a high temperature coefficient of electrical resistance, such

as platinum or tungsten. The filament is exposed to the gases in the evacuated space and cooled by them; the temperature, and therefore the resistance of the filament, depends on the heat conductivity of the gases, this in turn depends on their pressure. The filament forms one arm of a Wheatstone bridge, the compensating arm is an exactly similar wire sealed into an evacuated tube. The difference in electrical resistance between the two filaments, as measured by a sensitive galvanometer in the bridge circuit, indicates the gas pressure in the system.

THERMOCOUPLE GAUGE

This type of gauge, like the Pirani gauge, uses a filament with a high temperature coefficient of electrical resistance exposed to the gases in the vacuum system. The temperature of the filament, as measured by a small thermocouple connected to a sensitive millivoltmeter and attached to the heated filament, indicates the gas pressure. The range is 0·5 torr to 0·001 torr.

IONIZATION GAUGES

Ionization gauges measure the ion current produced by the passage of electrons from a heated wire to an anode maintained at a positive potential. The positive ions produced in the gas by collision with these electrons are attracted to a grid maintained at a negative potential. The ion current is a function of the gas pressure.

The length of the path of the electrons can be considerably increased by causing them to move in the field of a strong magnet, with consequent increase in ionization of the gas and greater sensitivity of the instrument.

The range of this type of vacuum gauge depends on the quality, and therefore the price, of the gauge head and associated electrical equipment. A moderately priced gauge will measure pressures within the range 10^{-2} torr to 10^{-5} torr. The most elaborate

and costly instrument is said to measure pressures as low as 10^{-11} torr.

The emitter, or heated wire, of an ionization gauge will be permanently damaged if it is exposed to gas pressures greater than 10^{-2} torr. Some means of measuring pressures of more than 10^{-2} torr should be provided.

Vacuum pressures of less than about 10^{-6} torr are generally considered to be in the ultra high vacuum range and are beyond the scope of this book.

Degassing

All glass and metal surfaces adsorb gases and vapours which are slowly released under vacuum. This process, known as degassing, continues for many hours at room temperature and is often mistaken for a leak in the system. Stopcock greases also degas at low pressures. Careful heating with a handlamp flame will speed up the degassing of glass tubes, but stopcocks should not be heated as the thin layer of grease will be upset. "Apiezon" greases should not be warmed to over 30°C.

The preliminary degassing of any new glass vacuum system may take more than 8 hr. If a number of stopcocks and ground glass joints are included and handlamp heating is inadvisable, the high frequency discharge from the leak tester can be used to help to release the adsorbed gases and vapours. Degassing can be speeded up by first cleaning all the glass components and annealing them in an oven. The assembly of the parts, either by ground glass joints or by handlamp joins, should proceed with the minimum delay. Every effort should be made to keep water vapour out of the system, and if it is necessary to admit air at any time, the air should be dried by passing it through a calcium chloride drying tube.

If the vacuum sought is close to the minimum pressure obtainable with the diffusion pump used, stopcocks and ground glass joints should not be employed on the high vacuum line. In any case the number of greased joints on the high vacuum side of any pumping system must be kept to the absolute minimum or

the ultimate vacuum will not be as low as that possible and the pressure will slowly increase when the manifold is isolated from the pump.

It is clear that some glassblowing skill is necessary when parts must be added to and removed from a high vacuum manifold from which stopcocks and joints are excluded by the nature of the project. The temporary attachments can be readily made by using 6–8 mm diameter tubing. Blowing can be done through a similar tube attached to the manifold, and cut open and resealed as required.

Planning a vacuum system

The planning of a vacuum system should be undertaken in consultation with the person who will use it, or with his supervisor. The type of work to be done with the apparatus and the ultimate vacuum required will determine whether a rotary oil pump or a diffusion pump must be used. The inclusion of a refrigerated trap or traps and the refrigerant to be employed will depend on the materials to be handled, and on their vapour pressures. The measurement of low pressures is not always necessary, but it is advisable to make provision for a vacuum gauge by including a side-arm to which the gauge may be attached should it prove essential at any later time.

The diameter of the glass tubes used for the components and connections in a system to be used at or above about 1 torr is unimportant, and 10 mm diameter glass will be satisfactory. Rubber tubing connections should be made with heavy-walled pressure tubing. It should be washed with 30% sodium hydroxide solution, rinsed with tap water then with distilled water, and, finally, dried in a desiccator. Rubber tube connections to glass or metal are made more easily if the tube ends are smeared with castor-oil or lightly coated with silicone grease. When less than 10^{-3} torr is sought, rubber tube must be avoided, or reduced to the minimum length. No rubber tube should be fitted to the high vacuum side of a diffusion pump.

VACUUM STOPCOCKS

Vacuum stopcocks are preferable to the ordinary quality as some selection of the latter will be required. Since the key bore will have some effect on the rate of evacuation, stopcocks of less than 3 mm bore should be avoided for the main vacuum line.

A metal desiccator fitted directly to the rotary oil pump inlet and provided with a vacuum release valve, a connection for a tilting McLeod gauge and one for the manifold or apparatus, is very convenient if pressures of not less than 10^{-3} torr are adequate.

SUPPORTS

Most glass vacuum systems need some type of support to prevent relative movement of the components. Special attention must be given to large stopcocks; considerable force may be required to turn them and inadequate support may easily result in breakage to the glassware.

Permanent or semi-permanent glassware can be supported with brass or galvanized iron saddles screwed to wooden blocks secured to a metal or wooden frame. Seasoned timber should always be used for frames as any shrinkage or other movement will endanger the glass. The saddles must be lined with resilient material to prevent any metal to glass contact, which can add to the possibility of breakage.

Laboratory clamps are useful for supporting glassware, they are readily adjusted when fixed to a framework of 12 mm ($\frac{1}{2}$ in.) diameter metal rods. Some care should be exercised to avoid introducing strains into the glass, either by clamping the tubes too tightly or by bending the tubes when the clamps are not quite in alignment. The bosshead screws should be tightened up after the clamp is in place, and should not produce appreciable movement of the glass.

The number of clamps or saddles used should not be more than will safely support the glassware. At least one should be placed near each large stopcock. In general small stopcocks do not require support.

Layout

A reasonably proportioned sketch of the vacuum system should be made and checked to ensure that no detail has been omitted. There must be sufficient space for a bunsen under the boiler of a gas heated diffusion pump. The refrigerated trap should be so located that a Dewar flask can be easily placed in position. Careful planning can reduce the number of stopcocks and ground glass joints, all of which should be accessible for manipulation and replacement. The general layout should be compact but not so cramped together that repairs and replacements to the glassware will be difficult. Small bore stopcocks, built into the main vacuum line, will not only reduce the effective speed of the pump but will also reduce the mechanical strength of the line. Mechanical strains tend to concentrate in the smallest diameter tube, where they are most likely to result in breakage. To sum up: a glass vacuum system should be efficient, robust and accessible.

The assembly of vacuum systems

All the component parts that can be made in the glassblower's workshop should be completed, washed and annealed. The stopcocks and other components that have been purchased as finished items are collected, cleaned and dried. Simple systems of compact design can be assembled by making the required bends with the bench burner and joining up the components with the handlamp. The glass is supported in a substantial retort stand and clamp, and arranged so that joins are made with the tubes involved in a vertical position. Some care must be taken to keep the joins straight and to avoid departures from vertical and horizontal lines, where such lines are desirable. An off-plumb cold trap or a manifold inclined to the horizontal will detract from the appearance of the apparatus. More elaborate systems can be made up into two or more parts. These parts are then mounted in position and joined together. Every effort possible

should be made to arrange matters so that this type of join is made in a vertical part of the glass line. If such an arrangement is impossible or inconvenient the horizontal join must be made with extra care to preserve the neatness of the system. When making joins between sections of a vacuum system it is advisable to set up that part which contains the diffusion pump and to support it adequately. Mercury diffusion pumps are heavy and a substantial base should be provided to take this weight. The section to be added is then placed in position and secured in such a way that the tube ends to be joined are almost touching. The supports on this part should now be loosened so that a small movement of the glass is possible. The join can now be made without the need for rod filler. Some rod should be at hand, however, ready for use if necessary.

When the join is made, and while the glass is still plastic, the supports are tightened. The join is then flame-annealed.

There is always a possibility that the handlamp flame will come in contact with woodwork, or with some part of the glass system other than the join. Such unintentional heating can cause damage and is best avoided by arranging that the joins are made in easily accessible positions, by covering nearby woodwork with asbestos board, and by wrapping stopcocks and other glassware, close to the join, with woven asbestos tape. These precautions are worth while. It is obvious that alterations made to the supports or clamps after the joins are made may result in substantial stresses in the glass, and that re-annealing of the joins will be desirable.

The Operation of a Vacuum System

The successful operation of any vacuum system is the result of experience and familiarity, rather than of instruction. The best results are usually obtained by an operator who has been using the system for some time; who knows the pressure conditions in any part of the vacuum line because he knows what has been done; who can estimate, from the sound of a rotary oil

pump, when it is time to bring the diffusion pump into operation, or whether there is a leak in the system or not. For example, an experienced neon tube processor can evacuate and degas the glassware and electrodes of such a tube, fill it with some 20 mmHg pressure of spectrally pure neon gas and seal the tube from the vacuum manifold, all in about 10 min. The only measurement he will make will be the pressure of the filling gas.

Obviously research projects cannot be carried out at this rate. Freeze drying, under vacuum, is a slow process and may take several hours.

References

1. Edwards High Vacuum Ltd., Manor Royal, Crawley, Sussex, England. Data sheet, B118/1.
2. Lloyd, J.T., A miniature McLeod gauge, *J. Inst. Sci. Techn.* **5**, 4 (1959).
3. Edwards High Vacuum Ltd., Manor Royal, Crawley, Sussex, England. Data sheet, D113/6.

CHAPTER 11

Interchangeable Ground-glass Joints, Stopcocks

PRIOR to 1930, and even later, many laboratory experiments were bedevilled with the glass apparatus available at that time. The component parts of the apparatus were, of necessity, connected together by corks and rubber stoppers; these connections were attacked by many chemical substances, particularly acids and solvents. Efforts to do work under reduced pressure also suffered and the purity of the products left much to be desired.

The introduction of ground-glass joints solved many of these problems; they were probably an extension of the simple and common ground-glass stoppers found in reagent bottles.

Interchangeable joints were the logical result of the increasing use of joints (Plate 11.1). The variations in proportions and dimensions found in the products of the different makers disappeared with the acceptance of standard specifications, and standard interchangeable ground-glass joints have become commonplace.

The value, to science, of these joints is very great indeed. Elaborate assemblages can be erected and dismantled in a very short time and the products from these all-glass assemblages are of a much higher standard of purity than those previously obtained. The excellent design of the glass components has resulted in a compact robustness undreamed of in the bored-cork and rubber-stopper era.

Conical joints consist of two members, the mating surfaces of which are both precision ground, and are part of a right circular cone whose vertical height is 10 times the diameter of the base (Fig. 11.1).

They are made by a number of companies in America, Britain

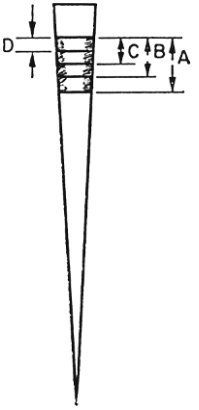

FIG. 11.1. Shows a standard taper conical surface with the height equal to ten times the diameter of the base. The ground surfaces of all standard taper conical joints are part of an exactly similar cone; the proportions of these surfaces depend on the purpose for which they are to be used and may be *A*, *B*, *C*, or *D*.

and Europe and conform to the accepted standard specifications of the country of origin. Each size of joint has a code number, the first part showing the diameter of the large end of the ground zone in millimetres or to the nearest whole millimetre; the second part showing the length of the ground zone in millimetres. Table 7 shows some codes for medium lengths as they appear in the makers' catalogues.

Medium length ground glass joints are by far the most popular. There are, however, some other lengths available for special purposes. Most glassblowers carry stocks of medium length joints and buy in other lengths as they are required.

Small sizes of joints are available with capillary tubes. The makers' catalogue should always be consulted and care taken to quote the catalogue numbers accurately.

British standard specification BSS572

American standard specification NBC C521

Standard interchangeable ground-glass joints are difficult to make in quantity. Some special equipment is essential. It can be argued that the money spent on such equipment could be spent to better purpose in buying joints from a reputable maker.

TABLE 7. INTERCHANGEABLE GROUND-GLASS JOINTS

British designation	Shortened form	American designation
5/13		5/12
7/16		7/15
10/19	0	10/18
12/21		12/18
14/23	1	14/20
19/26	2	19/22
24/29	3	24/25
29/32	4	29/26
34/35	5	34/28
40/38	6	40/35
45/40	7	45/50
50/42	8	50/50
55/44	9	55/50
60/46		60/40

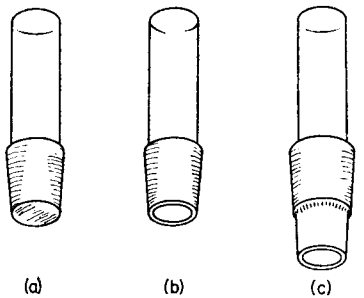

FIG. 11.2. Types of cones. (a) Closed-end cone, used for stoppers. (b) Open-end cone for general purposes. (c) Tube extension on cone, used for thermometer pockets, gas admission leads and other similar purposes.

Joint Design

Cones are made in three types. The first is closed at the small end of the ground zone and is used for stoppers. The second is open at the small end and is used for general purposes. The small end of the ground zone is extended as a tube in the third type; this tube may be cut off at an angle and is known as a drip tip (see Fig. 11.2).

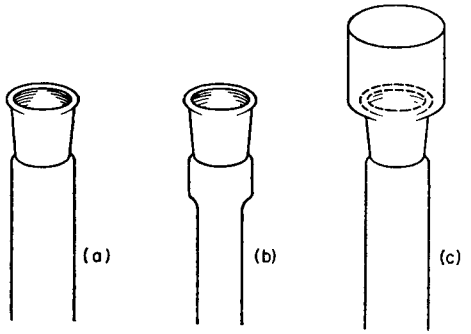

FIG. 11.3. Types of sockets. (a) Plain tube socket. (b) Socket with attached tube of similar diameter to that of the corresponding cone. (c) Cup socket.

Sockets are also made in three types, the first two differ only in the diameter of the tube extending beyond the small end of the ground zone. The third type has a short tube, or cup, attached to the large end (Fig. 11.3).

Making Joints

There are a number of methods employed in making interchangeable ground-glass joints. In some of these methods the cone blank is made to the specified length but slightly oversize in diameter to allow for reduction during grinding. The socket is made undersize in internal diameter, to allow for increase during grinding. Such blanks need not be accurately tapered. The grind-

ing of these blanks is done in two or more stages and is time consuming.

A description of a set of procedures that can be employed in a laboratory glassblowing workshop, and that involve a minimum of time spent in grinding, will be outlined.

EQUIPMENT

The following tools and equipment are essential: a glass-working lathe, one with a headstock chuck only will be sufficient. A set of standard taper hexagonal socket-forming tools made from carbon[1,2] (Fig. 11.4a). A set of standard taper moulds, also made from carbon[1,2] (Fig. 11.4b). These carbon tools are

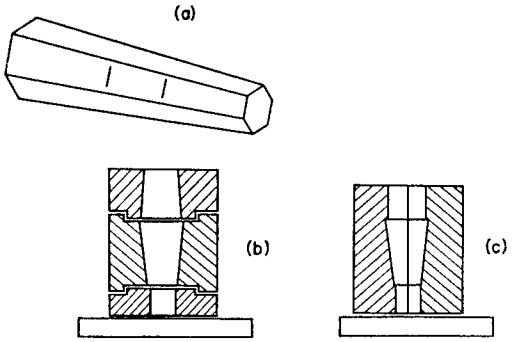

FIG. 11.4. (a) Socket-forming tool showing pencil marks indicating large and small end diameters and overall length of the socket taper. (b) A horizontally split mould. The sections are separated to remove the blown conical joint blank. (c) A vertically split mould. The two sections are separated when the joint blank is ready to be removed.

expensive and it is recommended that sizes smaller than 14/23 and larger than 34/35 be omitted. Great difficulty will be experienced in heating and controlling sufficient glass for such cones. The small sizes, i.e. 10/19 and 7/16, demand lengthy experience and above average skill to keep the glass wall-thickness

reasonably uniform. An attachment for the lathe, used to form the socket exterior, consists of a carbon paddle about 5 cm long, 6·25 mm thick and not less than the length of the ground

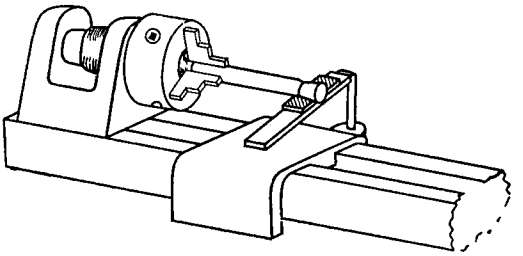

FIG. 11.5. Socket forming with a paddle fitted to a lathe.

zone +5 mm wide. This paddle is mounted on a hinged bar about 5 cm from the chuck jaws and inclined at 1 in 20 to the lathe centre line (Fig. 11.5). An adjustable stop can be improvised to control the elevation of the hinged bar.

TUBING

Although interchangeable ground-glass joints can be made from soda glass, special care must be taken to keep the formers and moulds hot. Some temperature just below the annealing temperature of the glass seems to be desirable.

Considerable freedom is possible when borosilicate glass is used. The author uses Pyrex brand for over 95% of the joints made by him.

The tubing should be of uniform wall-thickness and not less than 1·5 mm thick. Should the wall-thickness vary round the tube, the finished joints, especially the sockets, will not be symmetrical.

The glass for the sockets is cut into 25 cm lengths, so that a socket can be made on each end. The ends must be cut straight.

The tubing for the cones is prepared as in Fig. 11.6.

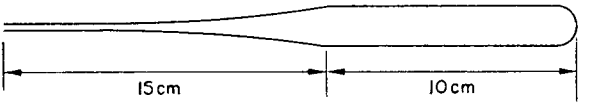

FIG. 11.6. A blank for a 14/23 cone.

TABLE 8

Socket size	Tubing diameter
14/23	17 mm
19/26	22 mm
24/29	27 mm
29/32	31 mm
34/35	37 mm

TABLE 9

Cone size	Tubing diameter
14/23	12 mm
19/26	17 mm
24/29	22 mm
29/32	27 mm
34/35	32 mm

SOCKET FORMING

If relatively small numbers of sockets are required at least twice that number should be made to allow for rejects. The tube is stoppered and mounted in the lathe; if the chuck has metal jaws, the glass is protected by a layer of asbestos paper.

The open end of the tube is heated with a soft flame from a handlamp fitted with a ribbon burner, which provides a flame about 2 cm wide and just hot enough to soften rather than melt the glass.

When the glass is plastic the tool bar is raised and the carbon

paddle pressed against the rotating glass to form a rough socket. Although, at this stage, the socket is rough, or imperfectly finished, it is important to have the internal diameter of the small end about 1 mm less than the finished size.

The socket is now reheated and the flame manipulated so that the edge gathers back into a thickened bead. The flame is removed when the glass is in a workable condition, and a previously selected and marked carbon tool inserted and gently pressed into the socket, until the small end mark on the tool coincides with the minimum diameter of the socket.

The procedure is easy to describe but usually takes some time to master. Should the glass be over-heated or unevenly heated, the application of the carbon paddle may cause the socket to be twisted. It is then beyond recovery and must be discarded. Should too much glass be heated or the carbon sheet be incorrectly adjusted the socket blank will again be distorted or of the wrong length. Should the glass be insufficiently hot for the first operation it will set before the rough formation is completed; if insufficiently hot for the second operation, it will set before the internal taper has been completely formed. The standard taper forming tool is not easy to use correctly. It must be held so that its centre line coincides with that of the rotating tube; otherwise the tool will flare the plastic glass instead of reaming it to required taper. The tool should be held lightly and allowed to move into the socket slowly so that the marks reach their required positions just as the glass sets. The tool should be withdrawn at once when the glass sets. Continued application of the standard-taper forming tool to rigid glass can only result in the tool being worn and the accurately machined edges will then be spoiled.

Cone moulding

The open fire-polished end of the point on the prepared tube is inserted into a length of small bore rubber tube; the other end of the rubber tube is connected to the foot- or manually-operated valve described in Chapter 4.

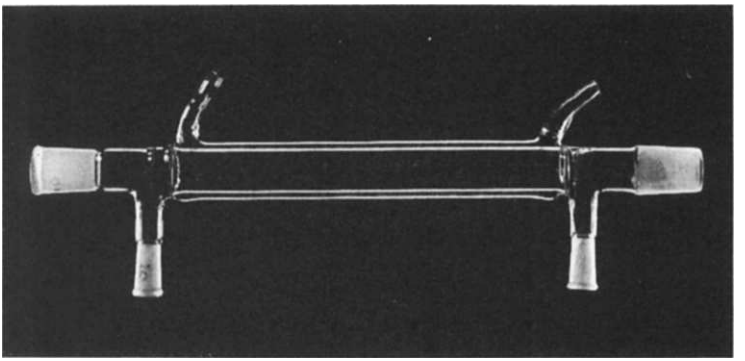

PLATE 11.1. An example of experimental glassware fitted with interchangeable ground-glass joints.

The point is held in the right hand, palm up, and rotated back and forth in a large flame. The heated glass is allowed to constrict and thicken, it is then lowered carefully into the mould and blown up. The cone blank is immediately removed from the mould and flame-annealed. The following details must be given attention: the blowing valve must be arranged so that expanding air in the closed tube can escape to atmosphere, otherwise the plastic glass will blow up prematurely and the tube will be spoiled. Sufficient glass must be heated to form the upper and lower shoulders and also the joint extension, if there is one. The blown cone blank must have a reasonably uniform wall-thickness, approximately the same as that of the original tube. If too much glass is melted, or if the plastic glass is allowed to elongate under gravity, the tube outside the mould will blow up. This is most undesirable with horizontally split moulds, as it involves waiting till the upper section is cool, then breaking the glass to remove it. With vertically split moulds the blown tube must be reworked to recover the original wall and diameter.

Grinding

Socket grinding

A case-hardened steel grinding tool of precise taper (Fig.11.7) is fitted to the chuck of an otherwise redundant lathe headstock and rotated at about 60 rev/min. A suspension of No. 400 silicon carbide (carborundum) in a mixture of glycerine and water is applied to the tool and the socket blank held in place with the right hand.

The glass is withdrawn every 10 turns or so to allow fresh carborundum to flow between the surfaces. The internal surface of the socket should be completely ground in about $1\frac{1}{2}$ min.

Prolonged grinding to correct defects in the shape of the blank must not be undertaken, as this will tend to wear the grinding tool.

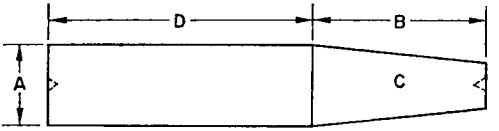

FIG. 11.7. A socket grinding tool. *A*, the diameter of the large end of the ground surface ±0·25 mm. *B*, the length of the ground zone +10 mm. *C*, standard taper of 1 in 10 in the diameter. *D*, any convenient length to allow for remachining, say 15 cm.

CONE GRINDING

The cones are ground into a similar accurately made cone grinding tool (Fig. 11.8) and again prolonged grinding must be avoided.

Carbon forming- and moulding-tools and case-hardened grinding tools must be provided for each size of joint being made. The standard taper surfaces of these tools must be protected from any type of wear or damage. The success of this method of making standard taper cones and sockets depends on the accuracy of the tools and on the accuracy of the glass blanks. Grinding must not be used to compensate for inaccurate blanks.

It should be understood that cone and socket blanks must be annealed, preferably in an oven, before the grinding is undertaken.

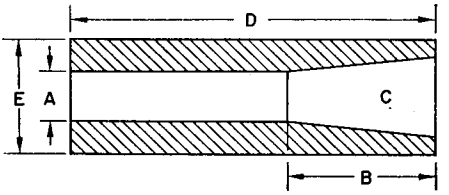

FIG. 11.8. A cone grinding tool. *A*, the diameter of the small end of the ground zone − 0·25 mm. *B*, the length of the ground zone +2 mm. *C*, standard taper of 1 in 10 in the diameter. *D*, about 15 cm to allow for remachining. *E*, any convenient diameter which allows an adequate thickness of metal.

The number of rejects from the first few batches of fifty cones and sockets may be as high as 50%.

There is a considerable possibility that the carbon tools will be damaged; they must be given careful treatment.

An air pressure of about 0·3 kg/cm^2 (5 lb/in^2) is required to blow up the moulded cones. Some provision should be made to keep the mould parts from separating when the contained plastic glass is exposed to this pressure. The horizontally split type can be weighted with a metal ring of suitable size. The vertically split type must be enclosed in a short length of metal tube, or in a vertically split and hinged container. These items can be designed and made in the engineering workshop.

Interchangeable ground-glass joints made by this method can be employed in apparatus used at ordinary pressures; some selection of the joints may be necessary if they are to be used for high vacuum work.

Ball and Socket Joints

A second type of ground-glass joint, known as a ball and socket joint, has appeared in recent years (Fig. 11.9). These joints are also known as spherical joints and as ball and cup joints. The two members are held together by a special spring-loaded clip. They provide limited flexibility to some types of

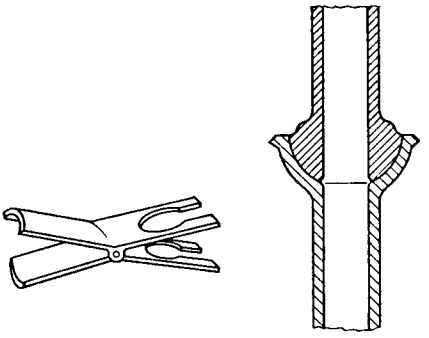

FIG. 11.9. Ball and socket joint with clip.

glassware and require rather less precise alignment than conical joints. Ball and socket joints must be robust and must have precisely spherical ground surfaces. The dimension tolerances are smaller than those permissible in conical joints and for this reason laboratory glassblowers should not undertake to make such joints. Ball and socket joints are given a code number in the makers' catalogues.[3] [4] This code number embraces the nominal bore of the tube and the dimensions of the cup or ball.

Flat Flange Joints

Flat flange joints are easier to dismantle than large diameter conical joints since they are less likely to seize under pressure or with prolonged contact at relatively high temperatures. Both

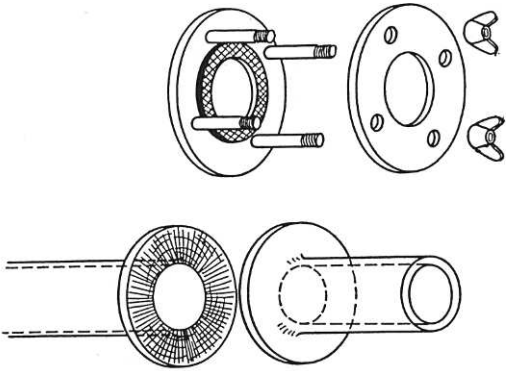

FIG. 11.10. Flat flange joint and clamp.

members of a union are similar and have flanges on the tube ends. The annular surfaces in contact with one another are ground flat and held together by metal clips designed for that purpose (Fig. 11.10).

Flat flanged joints can be made on tube of 25 mm diameter or less without the aid of special equipment. A lathe and an annealing oven are desirable when tubes of more than 25 mm are used.

The flat surfaces are ground by hand rotation on a sheet of plate glass. No. 400 carborundum suspension is used for finishing. If the blank surface is uneven and grinding must be prolonged, rough grinding may be necessary: No. 220 carborundum should be used.

All grinding procedures should be followed by thorough washing with running water. A firm test-tube brush will remove all traces of abrasive.

Stopcocks

All technicians are familiar with glass stopcocks, which are employed in so many items of laboratory apparatus.

The simplest types, shown in Fig. 11.11, provide a means of controlling and stopping the flow of gas or liquid through a tube. The barrel of a stopcock should be of robust construction, made from heavy-walled glass; the bore of the side-arms and of the key should be of the same diameter and precisely in line. The

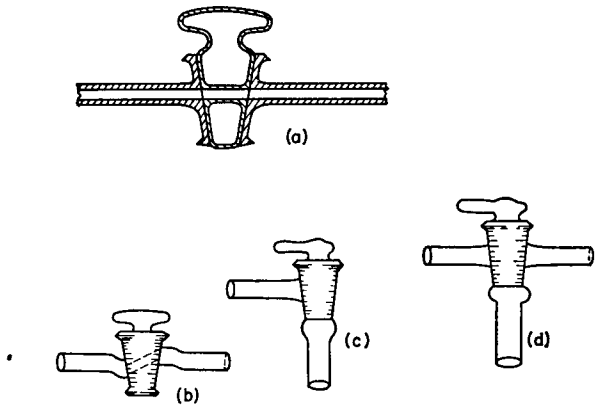

FIG. 11.11. Four common types of stopcock. (a) A straight stopcock with a hollow key. (b) An oblique bore stopcock with a hollow key. (c) An L-type stopcock. The key is a simple hollow cone with a hole drilled through it in line with the side-arm. (d) A T-type stopcock. The key is similar to that of (c) but the barrel has two side-arms. This stopcock provides alternative paths for gas flow.

junction between the side-arms and the barrel must be smooth and free from re-entrant angles. The side tubes should have reinforced wall-thickness extending a short distance from the barrel and tapering gradually to the thickness of the side-tube

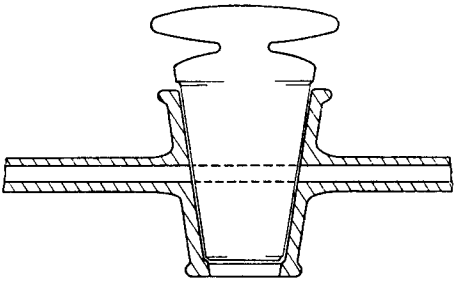

FIG. 11.12. The effect of wear on an imperfect stopcock. The shoulders on the key and barrel prevent the ground surfaces from making good contact with one another, and cause the stopcock to leak.

wall. The ground surfaces of the key and barrel must be of precisely the same taper and represent the same truncated cone, except that the large end of the key should not project beyond the barrel, and the small end of the key should project, by a small amount. The latter conditions ensure that any small change in dimensions, due to wear, will not produce a shoulder on the key and cause the stopcock to leak. (See Fig. 11.12.)

There are a number of different types of stopcock, each designed to serve a specific purpose. The cheaper types have solid keys, the key bore being drilled out. More expensive types have hollow blown keys, the bore or bores being small tubes joined across the key.

Stopcocks keys and barrels are not, in general, interchangeable, and some care must be taken to avoid confusing the keys when a number of stopcocks are being cleaned.

Vacuum stopcocks are more expensive than those intended for use at ordinary pressures. The ground surfaces are more precise and the makers test their products for vacuum tightness before offering them for sale. One type of vacuum stopcock,

shown in Fig. 11.11, has an oblique bore, which reduces the possibility of leakage through channels in the grease layer. The T-type and L-type, also shown in Fig. 11.11, are excellent for vacuum work. The keys of these types are held in place by atmospheric pressure when the apparatus is evacuated, and are therefore unlikely to be disturbed from their seatings when rotated.

The fabrication of stopcocks requires a high measure of skill and considerable glassblowing experience. Some expensive equipment is necessary for forming and grinding the keys and barrels. Most laboratory glassblowers are unlikely to have the time or opportunity to acquire such skills and the expenditure necessary to obtain the additional equipment will be difficult to justify. In general, stopcocks, and particularly vacuum stopcocks, should be purchased and built into the apparatus.

Elaborate stopcocks with more than two side-arms attached to the barrel are unsuitable for vacuum work. The keys of such stopcocks will have a very limited freedom of adjustment; and the path of potential leaks through the grease layer will be very short.

Lubricants

All ground glass surfaces should be perfectly clean and dry before the lubricant is applied in preparation for assembly. Dust in the joint may cause leakage, grit may cause breakage or may wear a small channel in the ground surfaces. These channels cause leakage which may be very difficult to trace. It is most unwise to rotate ground joints and stopcocks when they are dry, since a small piece of grit or glass will damage the surfaces.

The lubricant in a joint or stopcock serves to protect the surface from wear, to prevent leakage, and to facilitate the separation and rotation of the two members. Petroleum jelly is used for many assemblies but is unsuitable for equipment operating at high temperature or under vacuum. Some laboratories prepare special greases for use under conditions that do not permit hydrocarbons to be used.

"Apiezon" greases are suitable for ground joints and stopcocks. Three types are available: grease N is recommended for stopcocks and joints that will be rotated when under vacuum. Grease L is suitable for joints that are temporary and is recommended for refrigerated traps and P_2O_5 (phosphorus pentoxide) tube stoppers. A third type, M grease, is cheaper than the other two, and should be used when a moderate vacuum is required. Apiezon greases are suitable for vacuum work carried out at room temperature, but have an appreciable vapour pressure at more elevated temperatures.

Dow-Corning silicone stopcock-grease is resistant to a wide range of chemicals and stable from $-40\,°C$ to $200\,°C$. The vapour pressure is less than 10^{-6} torr and its consistency does not vary over a wide temperature range.

Silicone grease is not popular with glassblowers, however, as it is difficult to remove from glass surfaces, and can cause trouble in joining tubes that have been in contact with it. Instructions for its removal are printed on the container.

Lubricating joints and stopcocks

Both members should be clean and dry, and warmed to about $30\,°C$ in the burner flame or in an oven.

The grease is applied straight from the tube to the cone, or key, in a number of lines along the length of the ground surface. If the nozzle of the grease tube is removed from the glass when it is at or near the centre of the ground surface's length then a small blob of grease will be left there and will assist in expelling the air when the members of the joint are pressed together, without any turning.

The quantity of grease applied should be sufficient to provide a thin, uniform layer between the ground-glass surfaces. Excess grease will be squeezed from between the surfaces and may block the key bore or reduce its effective diameter. This grease will be wasted and, since vacuum grease is expensive, waste should be avoided. Insufficient grease is also undesirable. Air streaks are a

frequent cause of leakage and they occur when the layer is too thin or badly distributed. Insufficient grease has a further disadvantage in that it causes the stopcock key to be difficult to rotate and may sometimes result in breakage to the key handle.

Each time a ground glass assembly is dismantled the grease should be wiped off and renewed before reassembly; otherwise air pockets and streaks, both of which are undesirable, will almost certainly occur.

Clean tissue paper can be used to wipe off the grease, and pipe cleaners are useful for cleaning out small bore keys. When glassblowing work has to be done near any greased surface a more thorough cleaning than is possible with dry tissue paper will be necessary. Petroleum ether will remove most greases. Silicone grease can be removed by following the maker's instructions.

A clean matchstick can be used to apply grease from a jar to a ground surface. A moderately generous coating is applied to the conical surface and the surplus removed by stroking the glass, with the match, from the large to the small end. The grease is then left in small ridges separated by shallow channels along which the air can escape when the cone is pressed into the socket. It is possible to so manipulate the grease layer that rather more grease is left near the centre of the cone. This slight surplus will flow towards the ends, and help to expel air when the members are pressed together.

The following procedure is effective in preventing breakage when turning stopcock keys in their barrels. The barrel is held in the left hand and the key handle held in the right hand. The turning moment applied to the key is balanced, as nearly as possible, by an equal and opposite turning moment applied to the barrel. This simple method will reduce the possibility of breakage to the side tubes. If considerable force is required to turn the key the grease should be renewed without delay. All vacuum stopcocks should be opened, and closed, slowly.

Some care must be exercised to avoid overheating ground-glass surfaces when making joins. Should strain be introduced into precision-ground glassware, it will, from the definition of strain, be deformed, and the precision lost.

JOINING GROUND GLASS ACCESSORIES TO APPARATUS

Cones and sockets should, after cutting, have a length of tube left, not less than the length of the ground zone. The heating should not extend to the ground zone. The finished glassware should be oven-annealed.

Stopcock side tubes should be cut off so that a length not less than three times the diameter of the side tube is left.

Large stopcock barrels and ground glass joints are more easily strained than small ones and it is advisable that they be protected from excessive heat by wrapping them in woven asbestos tape. The key should always be removed from a stopcock during joining operations. The ends of the barrel can be plugged with asbestos stoppers.

Hooks are attached to joints if they have to support some weight or to withstand internal pressure above that of the atmosphere. The following procedure is recommended.

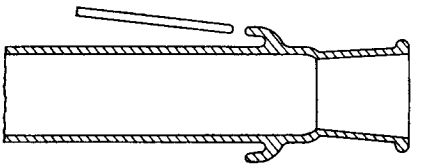

FIG. 11.13. Hooks on ground glass joints permit a pair of joints to be held together with springs or elastic bands. Re-entrant angles at the base of the hooks are undesirable and the result of insufficient melting of the glass.

The glass tube just below the joint shoulder is heated all round in a soft flame; the flame is then adjusted to a small sharp point and a spot on the joint tube heated. At the same time the end of a 5 mm rod is melted into a hot blob and this blob applied to the heated spot. The glass must be hot enough to make a good join. The rod is then pulled out, bent round parallel to the tube, and sealed off. A second hook is attached exactly opposite the first one (see Fig. 11.13). Unless the join is smooth, and the rod very slightly tapered, the hook will readily break off.

GROUND-JOINT HOLDERS

Accurately bored asbestos stoppers, carrying a suitable length of 8 mm tubing, make good holders. Discarded ground-joints can be used; they should have a suitable tube attached.

It sometimes happens that a ground-joint becomes detached from the holder during the glassworking operation. This is very inconvenient, especially if the work is being done in the lathe. A layer of asbestos paper will prevent this accidental separation, in most cases. Alternatively, the socket member of the union can be gently warmed before the pair are fitted together. The socket will then shrink and take a firm hold of the cone. They can be separated when required by gently heating the socket again and withdrawing the cone before the heat has penetrated to it.

Ground-glass holders are used by many glassblowers. The extra thickness of glass helps to prevent the ground surfaces being overheated and strained, or even deformed by melting.

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1. Morgan Crucible Co., Battersea Church Rd., London, S.W.11.
2. L. Richoux Co. (London) Ltd., 1965 Catalogue.
3. Quickfit & Quartz Ltd., Stone, Staffordshire, England.
4. Loughborough Glass Company Ltd., Loughborough, Leicestershire, England.

CHAPTER 12

The Organization of Glassblowing Classes

Elementary Classes

The preparatory work involved in conducting a glassblowing class must be undertaken a few months before the beginning of the teaching course.

The first essential is to engage an instructor who can combine the necessary standard of glassblowing skill, and knowledge of the properties of the materials used, with a sympathetic approach to the difficulties that will be met by the students.

The number of students that can be adequately supervised by an instructor is probably twelve. Each one will need some attention and special help. One hour of class time should be devoted to such help even if it lasts for only five minutes per student.

This work demands close attention to a number of details and is tiring for beginners; the class time should, therefore, be limited to about two hours.

The rate of progress of each student depends on his natural aptitude for glassblowing, on the teaching skill of the instructor, and on the time spent in practical work. It also depends on the frequency of the classes, so wherever possible they should meet at least twice a week. Students should be encouraged to practise the basic exercises in their own time; provided always that facilities for practice are available.

It is generally accepted that soda-glass tubing is the best material to use, for the following reasons: soda glass is cheaper then borosilicate glass, and fairly large quantities will be required. The techniques employed in working soda and borosilicate are very nearly the same, but although skill acquired in working soda

glass can be easily applied to borosilicate glass, it is not so easy to work soda glass if training has been given in borosilicate only.

Each student should be provided with 1·5 m (5 ft) of bench space, and a locker in which to keep his equipment. The bench should be fitted with a gas tap and a compressed air tap at the back of the bench, or under the front edge provided the taps and pipelines do not encroach on the knee space.

The locker should contain the following equipment:

- A bench burner, and flexible tube connections of convenient length.
- An asbestos sheet, about 30 × 30 cm, painted flat black or dark green.
- A substantial test-tube rack or wooden cooling-rack and a V-shaped tube-rest. Both these items can be made in the woodworking shop.
- Safety spectacles.
- A glass-cutting file or a glass knife.
- Four brass flanging-tools, a piece of beeswax or Nuboid.
- A pair of forceps, about 12 cm long.
- A selection of cork stoppers to fit the sizes of tubing in stock.
- A pair of outside calipers.
- A substantial retort stand with one or two bossheads and clamps.
- A 30 cm (12 in.) ruler.
- A glassmarking pencil.
- A suitable length of rubber blow-tube and some "policemen" (or rubber plugs).

Each student should bring a laboratory coat and should wear it throughout the classwork. He should provide himself with a notebook and should be encouraged to take notes and to make dimensioned sketches.

The instructor should make reasonably proportioned freehand sketches, of the glassware to be made, on the blackboard. The sketches should indicate the order of the steps to be taken.

The student should be encouraged to copy these drawings in his book.

The student should also bring a clean duster and should dust the tube stock before cutting it up for use.

The classroom should be provided with a set of cork borers and a sharpener, a glass-cutting machine with a rubber-bonded carborundum wheel, a roll of corrugated paper, some asbestos paper tape and asbestos cord, a diamond glass-cutter, any other special tools and accessories that the instructor thinks will be worth showing to his classes.

Handlamp Work

Should handlamp work be included in the course, a simple handlamp will be required for each locker. Alternatively, a general-purpose burner can be used. This burner is essentially a handlamp, but is supplied with a bench-mounting clamp and can also be used as a bench burner. The Flamemaster Mark II, made by Stone Chance Ltd., and obtainable from most laboratory furnishers, has a number of interchangeable flame-units and can be used with hydrogen, coal gas, natural gas or any of the liquid petroleum gases, together with compressed-air or oxygen. It is essential that the appropriate flame-unit, for the gas and oxidant used, be fitted.

Classwork

Classes should begin punctually and everything possible should be done to create an atmosphere of efficiency. The instructor should come to the class with a prepared plan of work. This should begin with a short lecture on the history of glass, and lead on to its use in modern science. A description of the most important types of glasses, their uses, and their relevant physical properties will help to capture the interest of the class members, and give them added knowledge of the material they are working with.

These lectures should be short, and the lecturer should provide examples of finished glassware and discuss their function. The care and use of the equipment supplied should be dealt with, preferably on the first day, when the burner is fitted up. Special attention should be given to fitting the rubber-tube connections to ensure that the students make these connections to the proper stopcocks.

The early part of the course should be devoted to basic operations, such as cutting straight ends on tubes, measuring lengths and diameters with reasonable accuracy, drawing straight strong points and making good joins. These basic operations should be combined so that the student has a definite goal in mind. If he sets off to make six test-tubes he will learn to measure diameters and lengths, to cut and rim straight ends on tubing and to blow round bottoms on the other end of the tubes. Similarly he should learn simple tube-bending by making sets of wash-bottle fittings and become competent at preparing and attaching side-arms by making side-arm test-tubes. He will also take more care when learning to make internal seals if he is making a saliva trap or a constant-level device.

After two or three months the students should be able to devote the full two-hour sessions to glassblowing practice, without showing signs of physical tiredness. The lectures should be completed by this time and the instructor should confine his activity to describing the exercise to be undertaken and to demonstrating the work. Most students learn glassblowing by copying the hand and finger positions of the instructor, and by imitating his movements. They tend to adjust the flame size and intensity so that they are the same as those used by the instructor. Consistent and frequently repeated demonstrations are most effective in teaching glassblowing.

The class members will divide themselves into three broad groups: those with better-than-average performance, average, and less-than-average performance.

The first group should be encouraged to improve the quality and quantity of their work, and, towards the end of the course, should be given more difficult tasks. The emphasis should be on improved quality rather than on greater complexity.

The last group must not be written off as hopeless. The instructor can help these students by repeating the early exercises over and over again, and by studying their techniques to find out if there are any faults that can be corrected. Faulty posture and improvised hand positions should be discouraged as soon as they are detected. A moderate discipline, imposed by the instructor at the beginning of the classwork, will be appreciated by the class and may save the instructor from the needless task of convincing his less co-operative pupils that his way is best.

All classwork should be tested by examination at least twice in the year. The examination should include one or two questions based on the lectures and designed to establish whether the students understand the variation in the physical properties of the different types of glasses. At least one question should cover thermal-expansion properties and annealing. Questions on practical work should embrace quantity and quality, and at least one should be included to test the skill of the better-than-average group.

A register of attendance will help to decide whether any students should be excluded from the final examination because of frequent absences. A record of class work will be useful to the instructor if he is called upon to establish that the full syllabus has been covered.

The apparent simplicity of some examination syllabuses should deceive neither the students nor the instructor. Every effort should be made to reach the highest possible standard in all the required aspects of the work. Examination nerves can play havoc with manual skills and, unless these skills are firmly established by long practice and frequent repetition, they may vanish or at least deteriorate when the student is under pressure.

Two or three class meetings should be devoted to revision work just before the examinations.

Handlamp work may be used as an introductory technique. Instruction and demonstrations can be given in making constrictions suitable for subsequent sealing off, either under vacuum or atmospheric pressure; and in making straight and T-joins.

These are useful skills for many laboratory technicians and it is regrettable that they are not given more attention.

Marking Examinations

The instructor should prepare model answers to the written questions, and allocate marks to all important parts of these answers. The marking must be consistent and absolutely impersonal. All superfluous answers must be ignored; otherwise the examiner may find that he has awarded a student more than 100% for his answers to one question.

EXAMPLE. *Question:* Where should a non-return gas valve be installed? What is its function? *Answer:* A non-return gas valve should be fitted to the principal gas-pipe; and should be so located that all the gas used in the glassblower's workshop passes through it. Should compressed air, or oxygen, enter the gas pipes for any reason so that the direction of the flow of gas is reversed, the non-return valve will close and prevent the formation of an explosive mixture of gas and oxidant in the pipe leading from the meter.

Two marks should be given if the student writes in his answer that the non-return valve is fitted to the principal gas pipe, one mark is given for each of the following parts of the answer: All the gas used in the workshop must pass through the valve. The entry of oxidant into the gas pipes creates an explosion hazard. The valve closes if the gas-flow is reversed. Explosive mixtures are confined to the workshop gas-pipe installation.

In the practical work marks should be given for every part of the exercise, for all dimensions that are within limits of error decided by the examiner, for straight cut and properly fire-polished, rimmed, or flared ends; credit should be given separately for even wall-thickness, uniform diameter, and accurate alignment of joins; blown bulbs should be assessed by their symmetry and wall-thickness.

Any attempt by the examiner to make an overall assessment of the student's level of achievement will only result in confusion, and can be unfair to students who do not succeed in completing some exercises but none the less make a workmanlike attempt at preparing the parts. Such preparation could be worth 50% of the marks allocated to that exercise.

Marks should be awarded for tidiness, economy in the use of glass tubing, and efficiency in setting out the equipment before the practical work begins and clearing everything away when it is finished.

The glass tubing and rod required for an elementary glassblowing course will depend on the personal choice of the instructor. The following list is set out as a guide.

			Standard packs
Rod	3·5 mm		1
	7·0 mm		1
Capillary tube	7·0 mm	1·25–2·00 mm bore	1
	9·0 mm	2·5–3·50 mm bore	1
Standard wall tubing	6 mm		1
	10 mm		1
	13 mm		1
	16 mm		1
	19 mm		1
	25 mm		1
	32 mm		1

Intermediate Glassblowing Classes

The formation of an intermediate class is warranted if sufficient numbers of elementary students reach the required standard of skill in performing the basic operations, and wish to proceed to a higher standard.

The work of such a class should be biased towards producing the more simple types of glassware used in the science laboratories. The emphasis should be on the quality and quantity of the items made, and on the economical use of the materials and accessories employed.

Intermediate glassblowing is important to those students who plan to put their skill to practical use. At this stage the opportunity is given to establish basic skills on a secure foundation; to acquire the ability necessary in making a large number of items, all very nearly identical in shape, all with neatly cut ends, adequately fire-polished, and all flame- or oven-annealed.

An improved range of glass tubing stock will be required. The variety of tubing diameters and wall-thicknesses will depend on the needs of the science laboratories. The instructor can be depended on to make an informed guess covering both the sizes and quantities that may be needed. If the source of supply of tubing is a considerable distance away, or even overseas, the quantities bought should be generous. The order should be placed about 6 months before the materials are to be used.

Most, if not all, of the items should be made of borosilicate glass and some stopcocks and ground-glass joints will be useful. The additional expenditure on stock will be partly offset by the value of the glassware made by the class.

The instructor should control requests for glassware made by the science departments, and prepare an instruction sheet for some items. A dimensioned sketch should be drawn on the sheet, together with details of the preparation of the component parts, and, where desirable, of the order of assembly of the parts.

The student should learn to make his own sketches and to make simple glassware to a sample.

Advanced Glassblowing Classes

The remarks set out under the previous heading also apply to advanced glassblowing, except that the standards sought are higher and the glassware made will be of greater complexity. For example, the students should learn to make double surface condensers of various patterns, and, if required, should prepare and assemble the components of the glassware for a vacuum line. It is also desirable that they receive some instruction in the use and maintenance of the vacuum apparatus.

The class should be given every possible opportunity to undertake repairs of all kinds, and to discuss, with the instructor, the procedures to be used.

The financial outlay required to maintain an advanced glassblowing class will depend on the needs of the associated science-teaching departments. If these departments are large and actively

engage in research work which requires glassware, the class could easily be an economic success.

Student numbers are unlikely to be large at this stage. None the less, a wide range of tubing sizes, of stopcocks and joints, will be required. Additional equipment, including at least one glass-blowing lathe, should be installed. A stock of silica tubes and accessories may have to be considered.

The instructor should encourage the students to use the library, to seek out and read all relevant literature. He should also encourage them to discuss the apparatus in hand with the scientist who has need of it; to follow up the completion of equipment by observing how it functions and what its end-products are.

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Appendix

Glass Vacuum Lines

Glass vacuum lines are designed to contain a range of active chemicals in the gaseous state without contamination and at low pressure. Glass tubing is, of course, used to make the various containing vessels and the tubes that connect those vessels together. Ground glass stopcocks were, until recently, used to isolate those vessels from one another as the need arose. Mercury was used as the manometric liquid for measuring the pressures within the vessels, and vacuum grease of various kinds was used to seal the vacuum stopcocks and ground glass joints in the system. The vacuum greases available have temperature limitations, they have a vapour pressure, however small, they absorb gases which are evolved at low pressure and none of them are inert. Stopcocks and joints have to be regreased at each reassembly, the grease has a limited life in use, and is now relatively expensive.

Efforts to eliminate grease from sensitive parts of vacuum lines have resulted in the use of a number of new materials with low vapour pressures, an extended working temperature range, and improved resistance to a number of chemicals. Three of the most important of these materials are described.

Teflon. This synthetic material is chemically inert, and has a safe working temperature of 120°C. It has a relatively high permeability to gases and is suitable for dynamic vacuum work, but is not recommended for static systems. Teflon (or P.T.F.E.) is a relatively deformable material, has a low coefficient of friction and is eminently suitable for making seals in stopcocks and taps.[1]

Teflon must not be heated to elevated temperatures as it decomposes, emitting a very poisonous gas.

Neoprene. Neoprene is an elastomer with mechanical properties very similar to natural rubber. It is manufactured by Du Pont and has a safe working temperature range of 15°C to 80°C. It is resistant to oils, to organic solvents, to some caustic solutions and to salts. It is not suitable for hydrogen peroxide or sulphur trioxide. Neoprene has low permeability to gases and is good for high vacuum and static vacuum systems.

Viton A. Manufactured by Du Pont, Viton A is also an elastomer, and has a safe working temperature range of −50°C to 250°C. It is resistant to many chemicals, to steam, ozone and fuming nitric acid, but not to ketones. Viton A has a low permeability to gases and is good for high vacuum and static vacuum systems.

"O"-rings. Both Neoprene and Viton A are usually supplied in a number of sizes of "O"-rings and are admirably suited to incorporation in vacuum taps and demountable joints.

"O"-ring taps and joints come in a variety of brands and designs, the more expensive types are usually more robust and have a long life.

Moisture trap attachments[2] for rotary oil immersion vacuum pumps and glass vacuum desiccators[3] have had impressive success when "O"-rings were fitted as vacuum seals.

Notes

A vacuum. In general terms a vacuum is any pressure less than atmospheric pressure, e.g. as in a vacuum cleaner or a milking machine.

In the laboratory:

A rough vacuum. Any pressure between atmospheric and that pressure produced by a water jet pump, i.e. down to about the vapour pressure of water, is known as a rough vacuum.

A vacuum. In vacuum line terms a vacuum is any pressure down to about 10^{-2} Torr.

A high vacuum. A pressure within the range 10^{-2} to 10^{-6} Torr is known as a high vacuum.

An ultra-high vacuum. The lowest range of pressures, lying between 10^{-6} and 10^{-9} Torr or better, is termed an ultra-high vacuum.

A dynamic system. This is a system designed to contain condensable gases and in which the ultimate vacuum, sometimes necessary for difficult fractional distillations, does not have to be maintained for prolonged periods of time.

A static vacuum system. Such a system is one in which the ultimate vacuum does have to be maintained for prolonged periods of time, say several days or, in some cases, months. Static vacuum conditions are required for the storage of refined products, but not necessarily for their preparation.

Units and measurement. Gas pressures of less than one atmosphere have been measured in a number of units, e.g. pounds per square inch, grams per square centimetre, and in SI units newtons per square metre or pascals.

In the laboratory, gas pressures have been measured with a manometer, or a McLeod gauge, using mercury as the manometric liquid, and the unit of such pressures has become "the pressure due to a column of mercury 1 mm high", usually written mmHg and now known as Torr.

In most vacuum work where a high degree of accuracy is not required, gas pressures are measured in Torr. Where published results call for the highest degree of accuracy, gas pressures measured in Torr can be corrected for temperature and gravity and converted to pascals using the definition

$$\text{Torr} \triangleq 133.322368 \text{ Pa}.$$

Safety. All glass vacuum line work is accompanied by hazards.

(1) The glassware may break as the result of excessive mechanical stress due to inadequate support for accessories, which are sometimes numerous and connected one to the other; to inadvertent movement of the line-supporting clamps; to inadequate greasing of stopcocks and unskilled attempts to turn the keys; to residual thermal stress caused by inadequate annealing techniques; to expansion of contained water, when frozen, and to confined gases expanding when the refrigerant is removed. These hazards can be

minimized by taking precautions when setting up the project.

(2) Any increase in pressure, whatever the cause, may be sufficient to blow ground glass joints apart, or to cause some part of the line to explode, usually that part of the line with the greatest diameter of tube.

Safety spectacles should always be worn by everyone in the vicinity of a glass vacuum line. When the chemicals handled in any vacuum line are known to be poisonous, the line should be in a fume cupboard.

Pressure release devices such as mercury manometers with one limb open to the atmosphere, or "O"-ring cup joints, should be included in all sections of a vacuum line which may be subject to excessive internal gas pressures.

(3) Many slush baths[4] are toxic—they should be prepared in a fume cupboard.

(4) Many slush baths[4] are inflammable—the normal precautions should be taken to avoid fire or explosion, i.e. no flames should be permitted in the vicinity of these inflammable slush baths.

(5) All bulbs and wide diameter vessels should be shielded in wire gauze cages, wrapped in cheesecloth, or covered with adhesive fibreglass tape.

(6) Mercury vapour is now recognized as a health hazard. The safe permissible limit of mercury vapour in air has been set by the National Institutes of Health, U.S.A. at 100 micrograms per m^3.

All laboratories with exposed mercury surfaces, such as in beakers used as the open limbs of manometers, must have good ventilation, particularly in colder climates where windows are usually shut and normal ventilation is often very poor.

Clean up all mercury spillages without delay.

Do not eat or keep food in rooms where there is mercury.

Mercury distillation is particularly hazardous. The apparatus should be set up in a fume cupboard, preferably over a large tray to contain any spillage.

(7) Glass vacuum lines should be supported on substantial frames so that the glassware is exposed to the minimum risk of breakage, either from cleaners' equipment or from accidental contact with trolleys or people passing nearby.

(8) Glass vacuum lines used for chemical synthesis have to be cleaned up to avoid contamination of one product by another. When cleaning cannot be done by acids or solvents then the line must be cut up into sections. It is considered to be both quicker and cheaper to replace all dirty glass other than stopcocks, taps and joints. These items are much too costly to discard and they should be cleaned.

Some Vacuum Line Accessories

A cooling-water flow indicator (Fig. A.1). This indicator performs a number of functions when connected to the outlet of the water jacket on a diffusion pump:

(1) One can tell at a glance whether the cooling water is flowing through the system.

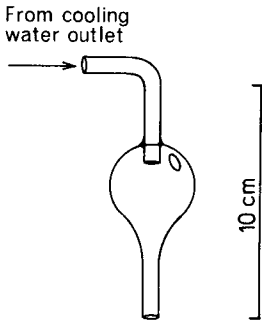

FIG. A.1. A cooling-water flow indicator. The drainage tube should be slightly larger in internal diameter than the inlet tube. The vent hole in the bulb ensures that the water pressure is independent of the length of the drainage tube.

(2) The device can be clamped in place, over the sink, eliminating the risk of floods that attend the use of a free moving rubber tube.

(3) Should a fail-safe device be used, of the type that switches off the power when the cooling water ceases to flow, then the water pressure in that device can be varied by raising or lowering the level of the indicator.

Note: The water pressure at the indicator is always atmospheric, in general the water pressure in the fail-safe device should be higher than atmospheric. A long length of tube pushed down the drain to reduce the risk of floods can lower the pressure in the device and make it ineffective.

Cajon "O"-ring metal connectors. Figure A.2 shows a sample collecting tube attached to a manifold by a Cajon connector. A very convenient arrangement for samples that do not react with metal.

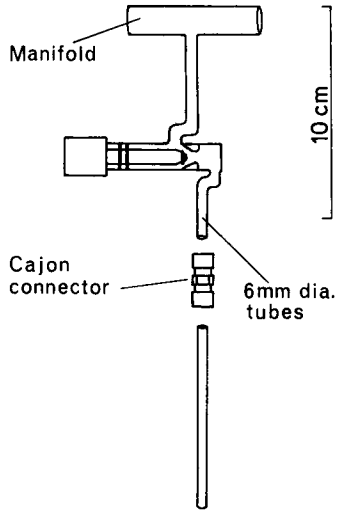

FIG. A.2. One application of Cajon "O"-ring metal connectors.

"O"-ring glass connectors. Two types are shown in Fig. A.3. They are used when the gases in a vacuum line may react with metals or grease. The double cup pattern can be used as a pressure release valve, a worthwhile precaution if there are no mercury manometers in the line.

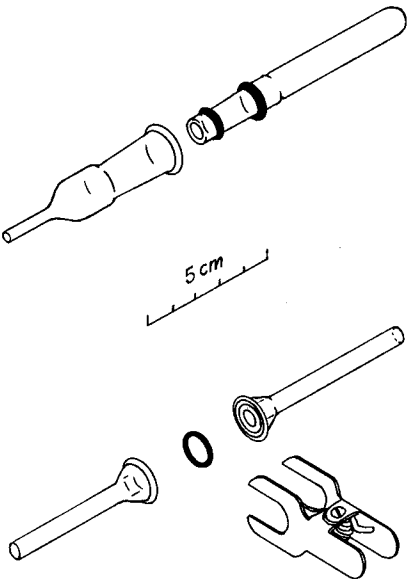

FIG. A.3. Two examples of glass "O"-ring connectors. The double cup pattern is held together with a spring loaded clip and is easily disconnected.

Screw thread connectors. These have many applications but are not suitable for high vacuum work. Two examples of these connectors are shown in Fig. A.4, with silicone rubber seals and a Teflon washer.

An H_2O separator (Fig. A.5). This separator is joined into a vacuum line and has a bypass tap. After the gas mixture has been condensed with liquid nitrogen through the tap on the left, the other two taps being closed, the separator is isolated from the

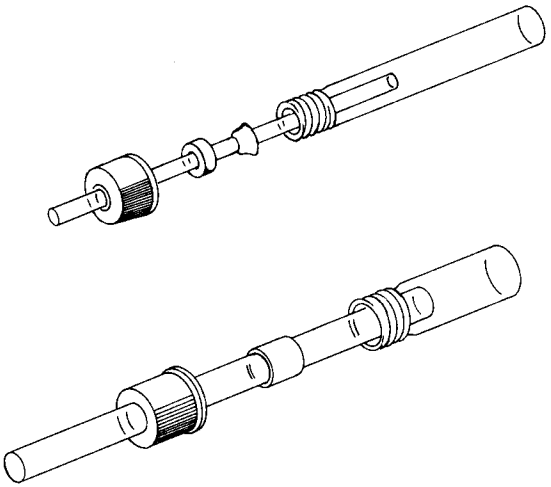

FIG. A.4. Two types of screw-thread connectors.

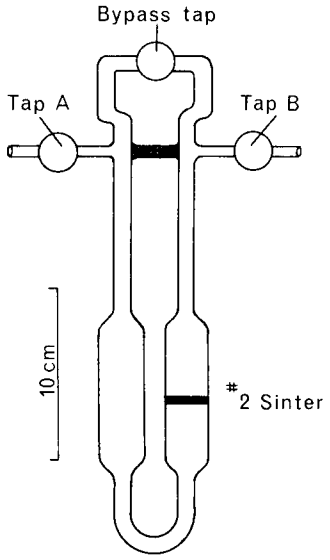

FIG. A.5. An H_2O separator with a No. 2, 10 mm diameter sintered disc. The U-tube and the sintered disc must be immersed in LN before the gas mixture is admitted to the separator.

line and the liquid nitrogen is replaced by a suitable slush bath. The H_2O is retained by the sintered disc, the other gases are transferred to another liquid nitrogen refrigerated trap through the open tap on the right.

The timing of the sequence of operations is important if good separation of water is to be achieved.

Another H_2O separator. This separator (Fig. A.6) is used when the gas and water mixture has to be transferred to another part of the vacuum line.

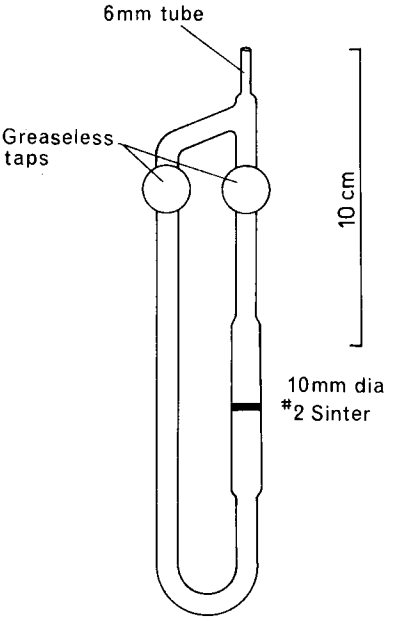

FIG. A.6. Another H_2O separator, this type is detachable.

A refrigerated impact separator. This pattern of separator (Fig. A.7) is used when the minimum resistance to gas flow is sought. Again timing is important. Refrigerants are used as in the separator shown in Fig. A.5. Water molecules striking the cold

outer tube adhere to the glass wall; other gases flow to the next refrigerated trap, only traces of water pass through.

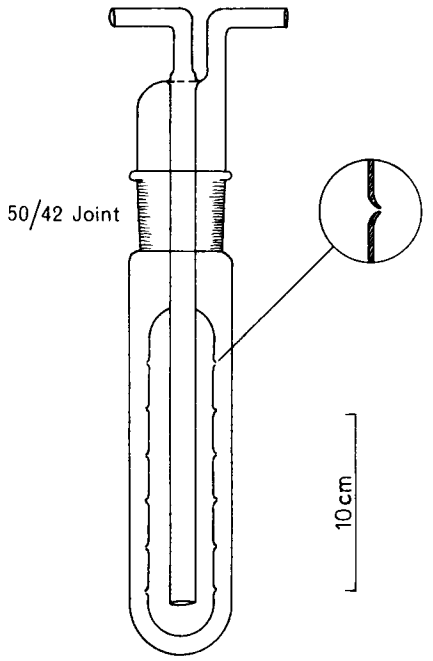

FIG. A.7. A refrigerated impact H_2O separator. The holes in the inner tube should be about 1.5–2 mm in diameter and distributed evenly over the cylindrical area.

A U-tube with greaseless taps. This is one type of collecting tube (Fig. A.8) for condensable gases. Some care must be taken when using this trap, and many other vacuum line accessories which can have condensable gases held within them. Should the refrigerant evaporate off, and both taps remain closed, then pressures from the enclosed gases may cause the glassware to explode. Safety glasses should be worn when operating any glass vacuum-line.

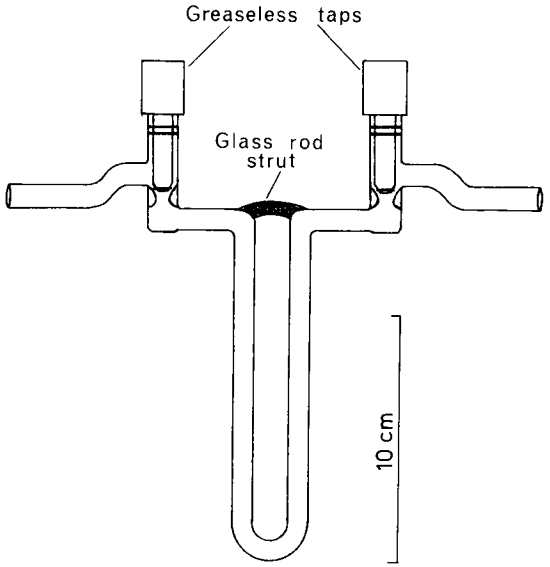

FIG. A.8. A U-tube for collecting condensable gases. Note that the lower limbs of the taps are connected to the U-tube so that potential leaks into the condensed gases are minimised.

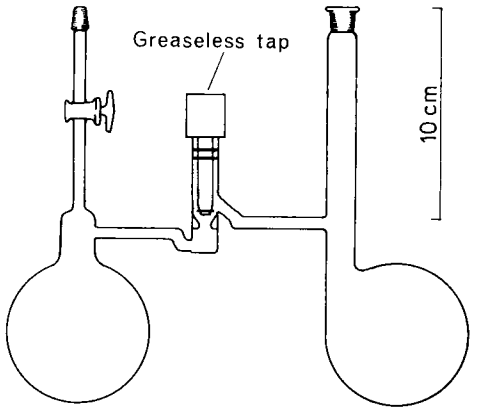

FIG. A.9. A pair of mixing vessels for air, grease, and water-sensitive reagents with a Teflon keyed tap.

Mixing vessels. Ideal mixing vessels for air, grease, and water sensitive reagents would be all glass, with no stoppers and no taps. They would, however, be difficult to evacuate, or to tilt or invert for transferring the reagents from vessel to vessel; there could also be problems with the isolation of products from residues. Sealing off would involve heating with perhaps product

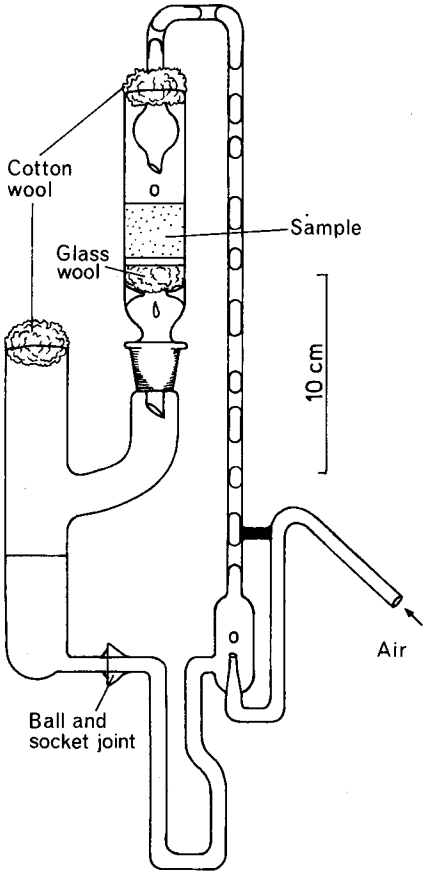

FIG. A.10. An improved perfusion unit. This unit operates with compressed air. The components are easily assembled and are interchangeable with those of similar units.

modification or destruction. The pattern shown in Fig. A.9 is, perhaps, an early step towards developing more versatile types of mixing vessels connected by inert taps.

An improved perfusion unit. Perfusion units of earlier design[5] used air-lift pumps operated by reduced pressure to circulate the aqueous solution. Such units are often used in banks of about ten and were difficult to adjust so that all of them circulated solution at the same rate. With this design, which uses compressed air, the air-lift pumps are easily adjusted and more positive in action. The solution can be readily sampled without dismantling the components. Additives to the solution can be introduced without disturbing the flow setting (Fig. A.10).

A break-seal attachment. This vacuum line attachment is used to recover samples sealed in relatively narrow tubes. The tubes are marked with a sharp glassknife so that, when they are inserted in the ground glass joint, passed through the stopcock key bore, and allowed to rest on the bottom of the envelope tube, the knife mark rests on the indentation in the envelope. After evacuation the crank is rotated slowly, as shown by the arrow in Fig. A.11. The sample tube will break cleanly at the knife mark.

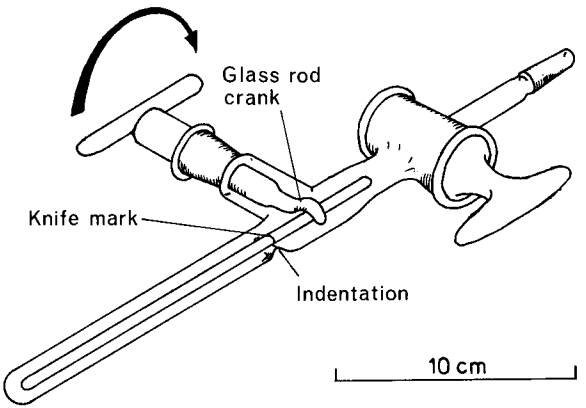

FIG. A.11. A break-seal attachment, used to open sealed N.M.R. tubes to allow for recovery of a sample.

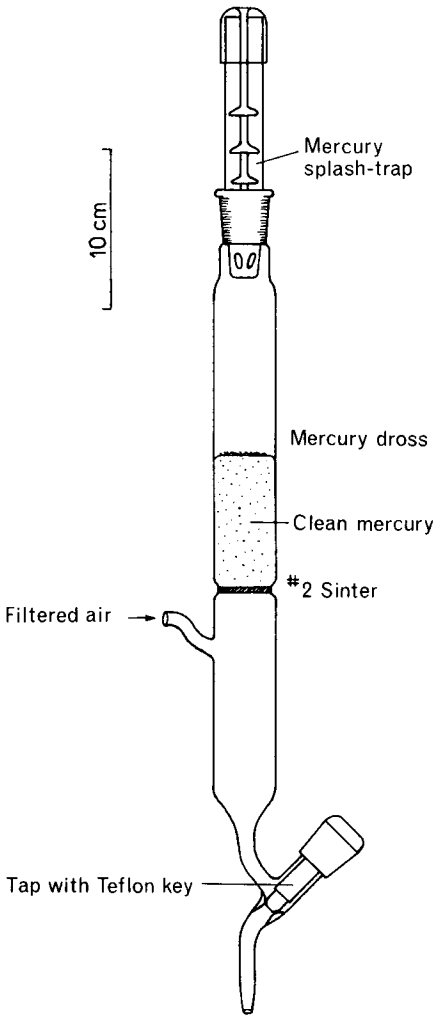

FIG. A.12. A device for cleaning mercury (Hg). This unit consists essentially of a No. 2, 30 mm diameter sintered disc filter tube with a Rotaflow tap on one end and a ground glass socket on the other. A Hg splash trap is incorporated in the stopper.

A device for cleaning mercury. Many vacuum lines use mercury manometers for pressure measurement and as pressure release safety devices. The mercury becomes contaminated by some compounds and tends to adhere to the manometer glass tube wall. The simple apparatus shown in Fig. A.12 will clean mercury so that it will not adhere to clean glass surfaces. The dirty mercury is poured into the space above the sintered disc through a funnel fitted with a filter paper having a small hole near its centre. A slow flow of glass-wool-filtered air is passed through the sinter

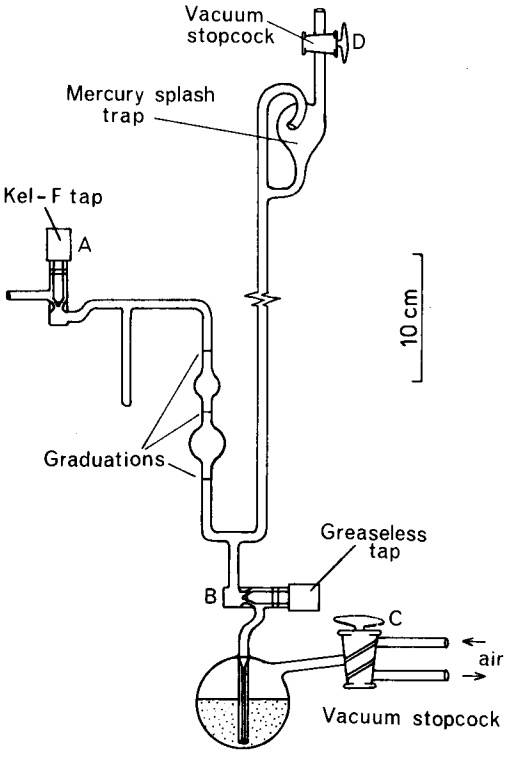

FIG. A.13. An improved constant volume manometer. The inclusion of a Kel-F type greaseless tap permits the measurement of grease sensitive gases. The Rotaflow tap between the U-tube and the reservoir prevents contamination of the Hg.

from below, so that the mercury is retained in the upper chamber and agitated by the air bubbles. After about 3 days the air flow is discontinued. All impurities will be oxidized. A further 3 days are allowed for all the impurities to float to the surface, then the mercury is filtered through the sinter. The mercury dross on the surface should not be allowed to contaminate the filter disc. The device can be cleaned with 10% nitric acid followed by tap water and distilled water. Should a higher standard of purity of mercury be necessary then distillation will be required.

A constant volume manometer.[7] This manometer is used to measure condensable gases quantitatively. It is not a new instrument, but its utility has been extended by the inclusion of two greaseless taps. The following notes are intended for those readers who are not familiar with the operational procedures (see Fig. A.13). The gas inlet tap A remains closed throughout the preparation and purification of the gas sample, and throughout the evacuation of the U-tube and bulbs. Tap A is a greaseless tap, preferably of the Kel-F type,[6] fitted with "O"-rings chosen to suit the gas being measured. Tap B will be exposed to mercury only, a Teflon tap such as Rotaflow, Fischer's, or Young's is recommended. Neither stopcock D nor stopcock C will be in contact with the sample gas, or mercury, and ground glass high vacuum stopcocks will be suitable.

The total capacity of the measuring bulbs should be chosen to suit the gas volumes to be determined and selected from a series of nominal capacities 5, 10, 25, 50 and 100 ml. The graduations between successive bulbs are best made before the tubes are joined up to the bulbs. The graduations are calibrated before the series of bulbs are built into the manometer.

The accuracy of a large capacity manometer can be improved by enclosing the series of bulbs in a water jacket and keeping the water at constant temperature.

The reservoir must contain sufficient mercury to fill all the manometer and still cover the open end of the capillary tube. The capillary tube in the reservoir prevents too rapid movement of the mercury. The constriction in the U-tube damps out oscillations of the mercury. The splash trap at the top of the mano-

meter reduces the possibility of mercury entering the manifold.

Calibration of the dead space above the top graduation mark. The space above the mercury in the reservoir is evacuated through stopcock C to about 10—15 Torr.

Tap B is slowly opened and the mercury level brought below the junction in the U-tube. B is closed. D is opened to the vacuum line manifold and the manometer pumped down to the lowest possible pressure. Ample time is allowed for drying and degassing. Tap D is closed. Some air is admitted to the reservoir through C and the mercury in the manometer allowed to rise to the graduation between the bulbs. Tap B is closed. The sample collecting tube is immersed in a liquid nitrogen bath and tap A opened to admit a small amount of pure dry gas, previously prepared. A is closed. The liquid nitrogen bath is removed and the gas sample allowed to warm up to ambient temperature.

By careful manipulation of tap B and stopcock C the mercury level in the manometer is adjusted to the graduation mark above the smallest bulb. The difference in the height of the mercury menisci in the limbs of the manometer is measured with a cathetometer and noted. The volume of the gas sample is now adjusted by manipulating B and C, so that the mercury meniscus is precisely at the graduation mark between the bulbs. Again the difference in mercury levels is measured and noted.

If v_1 is the volume of the dead space
v_2 the volume of the smaller bulb
p_1 the pressure of the gas at volume v_1
and p_2 the pressure of the gas at volume $v_1 + v_2$
then, assuming an ideal gas

$$p_1 v_1 = p_2 (v_1 + v_2)$$
$$\text{and } v_1 = \frac{p_2 v_2}{p_1 - p_2} \pm 2\%$$

To find the quantity of gas in a prepared sample. The entire sample is collected in the sample storage tube immersed in liquid nitrogen. The sample is then allowed to warm up to ambient temperature and its volume adjusted to one of the calibrated marks between the bulbs. The pressure, or difference in mercury levels is measured.

The quantity of gas is given by

$$q = pv = \frac{WRT}{M} \text{ in pressure volume units}$$

where p is the pressure of the sample, in pascals
v is the volume in cubic metres
W is the mass in kilograms
R is the universal gas constant in joules per kelvin per mole ($= 8.314\ \text{JK}^{-1}\ \text{mol}^{-1}$)
and T is the absolute temperature in K.

Manufacturers of Greaseless Accessories

The following list of manufacturers of greaseless taps, joints and connectors is taken from the glassblower's workshop catalogues. New Zealand offers a small market for such accessories and the list is probably nowhere near comprehensive.

(1) Edwards High Vacuum Ltd., Manor Royal, Crawley, Sussex, England.
(2) ACE Glass Incorporated, 1430 Northwest Boulevard, P.O. Box 688, Vineland, New Jersey, U.S.A.
(3) Cajon Company, 32550 Old South Miles Road, Cleveland, Ohio 44139, U.S.A.
(4) Kontes, Vineland, New Jersey, 08360, U.S.A. and The Elms, Kirklands Road, Over Killet, Lancs. LA6 1DP, England.
(5) J. Young, 11 Colville Road, Acton, London W3 8BS, England.
(6) G. Springham and Company Ltd., Temple Fields, Harlow, Essex, England.
(7) Fisher and Porter Co., Warminster, U.S.A.
(8) Quartz General Corporation, 12440 Exline Street, El Monte, California 91732, U.S.A.
(9) Quickfit and Quartz Ltd., Stone, Staffordshire, England.
(10) Jencons Scientific Ltd., Mark Road, Hemel Hempstead, Hertfordshire, England.
(11) Kimble Products, Owens Illinois, Toledo, Ohio 43601.

Refrigerants

The separation of condensable gases is achieved by cooling the mixture in a vessel attached to the vacuum line and immersed in liquid nitrogen (temperature −196°C); the gases are then allowed to warm very slowly and to pass through a series of traps immersed in refrigerants at known temperatures. Such refrigerants are prepared in dewar flasks by adding liquid nitrogen slowly to liquids of known freezing points until both solid and liquid phases are present. These partly frozen liquids are known as slush baths.

Of a range of liquids having freezing points from about 7°C to −180°C some are toxic and should only be used in a fume cupboard, and some are inflammable. Almost any temperature within the above range can be maintained by partially freezing suitable mixtures of liquids.

Slush baths.[4] The following slush baths are standard and used to achieve temperatures below 0°C.

CCl_4	Carbon tetrachloride	−23°C
C_6H_4Cl	Chlorobenzene	−45°C
$CHCl_3$	Chloroform	−63°C
CH_3CO_2Et	Ethyl acetate	−84°C
$C_5H_6CH_3$	Toluene	−96°C
n-C_3H_7OH	n-Propyl alcohol	−127°C

An example of a slush bath made with a mixture of liquids is carbon tetrachloride and chloroform 50/50 with a temperature of −81°C. Other proportions give other temperatures.

The well-known and relatively cheap mixture of ice, salt and water gives −20.1°C and solid CO_2 in acetone has a temperature of −80°C.

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